174

ANALYTICAL ABSTRACTS

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JOURNALS

The following list contains the names of journals (and their abbreviations) from which papers have been abstracted regularly during the year. Journals that have been omitted are those that contain only an occasional analytical paper and a number of Russian journals from which translated abstracts have been published through the medium of *Referativnŷĭ Zhurnal, Khimiya,* and *Referativnŷĭ Zhurnal, Khimiya, Biologicheskaya Khimiya.*

Abbreviated Title	FULL TITLE
Acad. R.P.R., Stud. Cercet. Chim.	Academia Republicii Populare Romîne, Studii si Cercetări de Chimie
Acta Chem. Scand.	Acta Chemica Scandinavica
Acta Chim. Acad. Sci. Hung.	Acta Chimica Academia Scientiarum Hungaricae
Acta Pharm. Jugoslav.	Acta Pharmaceutica Jugoslavica
Acta Polon. Pharm.	Acta Poloniae Pharmaceutica
Agric. Biol. Chem., Japan	Agricultural and Biological Chemistry [Japan]
Amer. Ind. Hyg. Ass. J.	American Industrial Hygiene Association Journal
Amer. J. Clin. Path.	American Journal of Clinical Pathology
Amer. J. Psychiat.	American Journal of Psychiatry
Anal. Biochem.	Analytical Biochemistry
Anal. Chem.	Analytical Chemistry
Anal. Chim. Acta	Analytica Chimica Acta
Analyst	Analyst
An. Asoc. Quím. Argentina	Anales de la Asociación Química Argentina
Angew. Chem.	Angewandte Chemie
Ann. Chim., Roma	Annali di Chimica [Rome]
Ann. Falsif.	Annales des Falsifications et de l'Expertise Chimique
Ann. Pharm. Franç.	Annales Pharmaceutiques Françaises
Ann. Rep. Agric. Hort. Res. Sta. Bristol	Annual Report of the Agricultural and Horticultural Research Station, Long Ashton, Bristol
Ann. Rep. Takamine Lab.	Annual Report of Takamine Laboratory
An. Real. Soc. Esp. Fís. Quím., B	Anales de la Real Sociedad Española de Física y Química, Serie B [Madrid]
Antibiot. & Chemother.	Antiobiotics & Chemotherapy
Antibiotiki	Antibiotiki [Moscow]
Appl. Spectroscopy	Applied Spectroscopy
Arch. Biochem. Biophys.	Archives of Biochemistry and Biophysics
Arch. Eisenhüttenw.	Archiv für das Eisenhüttenwesen
Arch. Pharm., Berlin	Archiv der Pharmazie und Berichte der deutschen pharmazeutischen Gesellschaft
Arzneimittel-Forsch.	Arzneimittel-Forschung
Atomnaya Energiya	Atomnaya Energiya [Moscow]
Aust. J. Exp. Biol. Med. Sci.	Australian Journal of Experimental Biology and Medical Science
B.C.I.R.A. Journal	B.C.I.R.A. Journal [British Cast Iron Research Association]
Ber. dtsch. keram. Ges.	Berichte der deutschen keramischen Gesellschaft
Biochem. J.	Biochemical Journal
Biochim. Appl.	Biochimica Applicata [Parma e Roma]
Biochim. Biophys. Acta	Biochimica et Biophysica Acta
Biokhimiya	Biokhimiya [Moscow]
Biul. Inst. Roślin Leczniczych	Biuletyn Instytutu Roślin Leczniczych [Poznan]
Boll. Lab. Chim. Provinciali	Bollettino dei Laboratori Chimici Provinciali [Bologna]
Boll. Soc. Ital. Biol. Sper.	Bollettino della Societa Italiana di Biologia Sperimentale
Brauwissenschaft	Brauwissenschaft
BrennstChemie	Brennstoff-Chemie
Brit. J. Appl. Phys.	British Journal of Applied Physics
Brit. Med. J.	British Medical Journal
Bull. A.S.T.M.	Bulletin of the American Society for Testing Materials
Bull. Cent. Belge Étud. et Docum. Eaux	Bulletin du Centre Belge d'Étude et de Documentation des Eaux
Bull. Chem. Soc. Japan	Bulletin of the Chemical Society of Japan
Bull. Inst. Chem. Res., Kyoto Univ.	Bulletin of the Institute for Chemical Research, Kyoto University [Japan]
Bull. Res. Council Israel	Bulletin of the Research Council of Israel
Bull. Soc. Chim. Belg.	Bulletin des Sociétiés Chimiques Belges
Bull. Soc. Chim. Biol.	Bulletin de la Société de Chimie Biologique
Bull. Soc. Chim. France	Bulletin de la Société Chimique de France
Canad. J. Biochem. Physiol.	Canadian Journal of Biochemistry and Physiology
Canad. J. Chem.	Canadian Journal of Chemistry
Cereal Chem.	Cereal Chemistry
Českosl. Farm.	Československá Farmacie
Chem. Age	Chemical Age
Chem. Anal., Warsaw	Chemia Analityczna [Warsaw]

Abbreviated Title	FULL TITLE
Chem. Ber.	Chemische Berichte
ChemikerZtg	Chemiker-Zeitung
Chem. & Ind.	Chemistry & Industry
Chemist Analyst	Chemist-Analyst
Chem. Listy	Chemické Listy [Prague]
Chem. Pharm. Bull., Japan	Chemical & Pharmaceutical Bulletin [Japan]
Chem. Průmysl	Chemický Průmysl [Prague]
Chem. Tech., Berlin	Chemische Technik [Berlin]
Chem. Weekbl.	Chemisch Weekblad
Chem. Zvesti	Chemické Zvesti [Bratislava]
Chim. Anal.	Chimie Analytique
Chim. e Ind.	Chimica e l'Industria [Milan]
Chimia	Chimia [Switzerland]
Clin. Chem.	Clinical Chemistry [Baltimore, U.S.A.]
Clin. Chim. Acta	Clinica Chimica Acta
Coll. Czech. Chem. Commun.	Collection of Czechoslovak Chemical Communications
Compt. Rend.	Comptes Rendus Hebdomadaires des Séances de l'Académie des Sciences
Compt. Rend. Acad. Bulg. Sci.	Comptes Rendus de l'Académie Bulgare des Sciences
Curr. Sci.	Current Science [India]
Dansk Tidsskr. Farm.	Dansk Tidsskrift for Farmaci
Diagn., Lab. e Clin.	Diagnosi, Laboratorio e Clinica [Naples]
Dissert. Abstr.	Dissertation Abstracts
Dissert. Pharm., Krakow	Dissertationes Pharmaceuticae [Krakow]
Drug Standards	Drug Standards
Dtsch. ApothZtg	Deutsche Apotheker-Zeitung
Dtsch. LebensmittRdsch.	Deutsche Lebensmittel-Rundschau
Endocrin. & Metabolism	Endocrinology & Metabolism
Energia Nucleare	Energia Nucleare [Milan]
Enzymologia	Enzymologia [Den Haag]
Erdöl u. Kohle	Erdöl und Kohle
Ernährungsforschung	Ernährungsforschung
Experientia	Experientia [Basle]
Farbe u. Lack	Farbe und Lack
Farmaco, Ed. Prat.	Farmaco, Edizione Pratica
Faserforsch. u. Textiltech.	Faserforschung und Textiltechnik
Fette, Seif., Anstrichmitt.	Fette, Seifen, Anstrichmittel
Food Res.	Food Research
Fuel, London	Fuel [London]
Geochim. et Cosmoch. Acta	Geochimica et Cosmochimica Acta [London]
Getreide u. Mehl	Getreide und Mehl
Grasas y Aceites	Grasas y Aceites [Seville]
Helv. Chim. Acta	Helvetica Chimica Acta
Hilger J.	Hilger Journal
Hoppe-Seyl. Z.	Hoppe-Seyler's Zeitschrift für Physiologische Chemie
Hutn. Listy	Hutnické Listy [Prague]
Ind. Chem.	Industrial Chemist
Indian J. Pharm.	Indian Journal of Pharmacy
Inf. Quím. Anal.	Información de Química Analitica [Madrid]
Inst. Hierro y Acero	Instituto del Hierro y del Acero [Madrid]
Int. Dairy Congr., Proc.	International Dairy Congress, Proceedings
Int. J. Appl. Radiation and Isotopes	International Journal of Applied Radiation and Isotopes
Int. Sugar J.	International Sugar Journal
Ital. J. Biochem.	Italian Journal of Biochemistry
Izv. Akad. Nauk SSSR	Izvestiya Academii Nauk SSSR
Izv. Vŷssh. Ucheb. Zavedenii, Khim. i Khim. Tekhnol.	Izvestiya Vŷsshykh Uchebnykh Zavedenii, Khimiya i Khimicheskaya Tekhnologiya
J. Agric. Chem. Soc. Japan	Journal of the Agricultural Chemical Society of Japan
J. Agric. Food Chem.	Journal of Agricultural and Food Chemistry [Washington, D.C.]
J. Amer. Chem. Soc.	Journal of the American Chemical Society
J. Amer. Leath. Chem. Ass.	Journal of the American Leather Chemists' Association
J. Amer. Oil Chem. Soc.	Journal of the American Oil Chemists' Society
J. Amer. Pharm. Ass., Sci. Ed.	Journal of the American Pharmaceutical Association, Scientific Edition
J. Amer. Wat. Wks Ass.	Journal of the American Water Works Association
Japan Analyst	Japan Analyst
J. Appl. Chem.	Journal of Applied Chemistry [London]
J. Ass. Off. Agric. Chem.	Journal of the Association of Official Agricultural Chemists
J. Biol. Chem.	Journal of Biological Chemistry
J. Chem. Educ.	Journal of Chemical Education
J. Chem. Soc.	Journal of the Chemical Society

Abbreviated Title	FULL TITLE
J. Chem. Soc. Japan, Ind. Chem. Sect.	Journal of the Chemical Society of Japan, Industrial Chemistry Section
J. Chem. Soc. Japan, Pure Chem. Sect.	Journal of the Chemical Society of Japan, Pure Chemistry Section
J. Chromatography	Journal of Chromatography
J. Clin. Path.	Journal of Clinical Pathology
J. Dairy Res.	Journal of Dairy Research
J. Dairy Sci.	Journal of Dairy Science
J. Electroanal. Chem.	Journal of Electroanalytical Chemistry
J. Indian Chem. Soc.	Journal of the Indian Chemical Society
J. Inst. Brewing	Journal of the Institute of Brewing
J. Inst. Fuel	Journal of the Institute of Fuel
J. Instn Chem., India	Journal and Proceedings of the Institution of Chemists [India]
J. Inst. Petrol.	Journal of the Institute of Petroleum
J. Iron St. Inst.	Journal of the Iron and Steel Institute
J. Lab. Clin. Med.	Journal of Laboratory and Clinical Medicine
J. Med. Lab. Technol.	Journal of Medical Laboratory Technology
J. Opt. Soc. Amer.	Journal of the Optical Society of America
J. Pharm. Pharmacol.	Journal of Pharmacy and Pharmacology
J. Pharm. Sci.	Journal of Pharmaceutical Science
J. Pharm. Soc. Japan	Journal of the Pharmaceutical Society of Japan
J. Polarographic Soc.	Journal of the Polarographic Society
J. prakt. Chem.	Journal für praktische Chemie
J. Res. Nat. Bur. Stand.	Journal of Research of the National Bureau of Standards
J. S. Afr. Chem. Inst.	Journal of the South African Chemical Institute
J. Sci. Food Agric.	Journal of the Science of Food and Agriculture
J. Sci. Ind. Res., India (B, C or D)	Journal of Scientific and Industrial Research [India], Sections B, C and D
J. Sci. Instrum.	Journal of Scientific Instruments
J. Sediment. Petrol.	Journal of Sedimentary Petrology
J. Text. Inst., Proc.	Journal of the Textile Institute, Proceedings
J. Wat. Pollut. Control Fed.	Journal of the Water Pollution Control Federation
Kem. u Ind., Zagreb	Kemija u Industriji [Zagreb]
Klin. Wochschr.	Klinische Wochenschrift
Lab. Delo	Laboratornoe Delo [Moscow]
Lab. Practice	Laboratory Practice
Lancet	Lancet
Magyar Kém. Foly.	Magyar Kémiai Folyóirat
Magyar Kém. Lapja	Magyar Kémikusok Lapja
Mater. Res. & Stand.	Materials Research and Standards
Mém. Poudres	Mémorial des Poudres [Paris]
Metallurgia, Manchr	Metallurgia [Manchester]
Microchem. J.	Microchemical Journal
Mikrochim. Acta	Mikrochimica Acta [Vienna]
Mitt. Lebensmitt. Hyg., Bern	Mitteilungen aus dem Gebiete der Lebensmitteluntersuchung und Hygiene [Bern]
Nahrung	Nahrung
Nature	Nature [London]
Naturwissenschaften	Naturwissenschaften
Olii Min.	Olii Minerali, Grassi e Saponi, Colori e Vernici
Optika i Spektroskopiya	Optika i Spektroskopiya
Öst. ChemZtg	Österreichische Chemiker-Zeitung
Peint.-Pigm.-Vern.	Peintures-Pigments-Vernis
Perfum. Essent. Oil Rec.	Perfumery and Essential Oil Record
Pharm. Acta Helv.	Pharmaceutica Acta Helvetiae
Pharm. Weekbl.	Pharmaceutisch Weekblad
Pharm. Zentralh.	Pharmazeutische Zentralhalle für Deutschland
Photoelect. Spectr. Gr. Bull.	Photoelectric Spectrometry Group Bulletin
Plaste u. Kautsch.	Plaste und Kautschuk
Plast. Massy	Plasticheskie Massy
Plating	Plating
Proc. Amer. Soc. Brew. Chem.	Proceedings. American Society of Brewing Chemists
Průmysl Potravin	Průmysl Potravin [Prague]
Pure & Appl. Chem.	Pure and Applied Chemistry
Radiokhimiya	Radiokhimiya
R.C. Ist. Sup. Sanit.	Rendiconti Istituto Superiore di Sanità
Rec. Trav. Chim. Pays-Bas	Receuil des Travaux Chimiques des Pays-Bas
Rep. Invest. U.S. Bur. Min.	Report of Investigations. U.S. Bureau of Mines
Rev. Asoc. Bioquím. Argentina	Revista de la Asociación Bioquímica Argentina
Rev. Chim., Bucharest	Revista de Chimie [Bucharest]
Rev. Fac. Ing. Quím., Argentina	Revista de la Facultad de Ingeniería Química [Argentina]
Rev. Métall.	Revue de Métallurgie
Rev. Sci. Instrum.	Review of Scientific Instruments

Abbreviated Title	FULL TITLE
v. Soc. Quím. Mex.	Revista de la Sociedad Química de Mexico
v. Univ. Ind. Santander, Colombia	Revista de la Universidad de la Industrial de Santander [Colombia]
c. Sci.	Ricerca Scientifica
echstoffe u. Aromen	Riechstoffe und Aromen
v. Ital. Sostanze Grasse	Rivista Italiana delle Sostanze Grasse
ɔczn. Zakł. Hig., Warsaw	Rocznik Państwowego Zakładu Higieny [Warsaw]
Afr. Ind. Chem.	South African Industrial Chemist
Afr. J. Agric. Sci.	South African Journal of Agricultural Science
and. J. Clin. Lab. Invest.	Scandinavian Journal of Clinical & Laboratory Investigation
ience	Science
i. Pharm.	Scientia Pharmaceutica [Vienna]
oil Sci.	Soil Science
ɔectrochim. Acta	Spectrochimica Acta
ud. Cercet. Chim., Cluj	Studii şi Cercetări de Chimie [Cluj]
uomen Kem.	Suomen Kemistilehti [Helsinki]
ensk Farm. Tidskr.	Svensk Farmaceutisk Tidskrift
ensk Papp-Tidn.	Svensk Papperstidning [Stockholm]
alanta	Talanta
APPI	TAPPI
extilrdsch	Textil-Rundschau
rans. Brit. Ceram. Soc.	Transactions of the British Ceramic Society
rudỹ Inst. Metallurgiĭ, Akad. Nauk SSSR	Trudỹ Instituta Metallurgiĭ, Akademiya Nauk SSSR
rudỹ Khim. i Khim. Tekhnol.	Trudỹ po Khimii i Khimicheskoĭ Tekhnologii, Nauchno-Issledovatel'skiĭ Institut Khimii Gosudarstvennogo Universiteta im N.I. Lobachevskago
rudỹ Komiss. Anal. Khim., Akad. Nauk SSSR	Trudỹ Komissii po Analiticheskoĭ Khimii, Akademiya Nauk SSSR
rudỹ Ural'sk. Politekh. Inst.	Trudỹ Ural'skogo Politekhnicheskogo Instituta
ɔh. Zap. Leningrad. Gos. Univ.	Uchengy Zapiski Leningradskogo Gosudarstvennogo Ordena Lenina Universiteta
kr. Khim. Zhur.	Ukrainskiĭ Khimicheskiĭ Zhurnal
.S. Atomic Energy Comm.	United States Atomic Energy Commission. Reports
.S. Geol. Surv. Profess. Papers	United States Department of the Interior, Geological Survey. Professional Papers
zbek. Khim. Zhur.	Uzbekskiĭ Khimicheskiĭ Zhurnal
ɜstn. Leningr. Univ., Ser. Fiz. i Khim.	Vestnik Leningradskogo Universiteta, Seriya Fiziki i Khimii
ɜstn. Moskov. Univ., Ser. Khim.	Vestnik Moskovskogo Universiteta, Seriya Khimiya
at. & Sewage Wks	Water & Sewage Works
anal. Chem.	Zeitschrift für analytische Chemie
avod. Lab.	Zavodskaya Laboratoriya [Moscow]
Chem.	Zeitschrift für Chemie
ɯur. Anal. Khim.	Zhurnal Analiticheskoĭ Khimii
ɯur. Prikl. Khim.	Zhurnal Prikladnoĭ Khimii
LebensmittUntersuch.	Zeitschrift für Lebensmittel-Untersuchung und -Forschung

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ostract Bulletin of the Institute of Paper Chemistry
ostracts of Bulgarian Scientific Literature
emical Abstracts
el Abstracts and Current Titles
tallurgical Abstracts
uclear Science Abstracts
utrition Abstracts and Reviews
ferativnyĭ Zhurnal, Khimiya
ferativnyĭ Zhurnal, Khimiya, Biologicheskaya Khimiya
view of Current Literature relating to the Paint, Colour, Varnish and Allied Industries
ubber Abstracts
irley Institute. Summary of Current Literature
gar Industry Abstracts
ater Pollution Abstracts

ERRATA

VOL. 2, 1955

Index, p. cxix, column 2, third line from bottom, *for* 3520 *read* 3529

VOL. 6, 1959

Abstract No.	*Line*
4775	2, 6, *for* dithbaiocarmoylhydrazinium *read* dithioformylhydrazinium

VOL. 7, 1960

Index, p. lxvi, column 2. The abstract number in the entry under **King. J.** should be 2416, not 3416. The abstract following 2415 is wrongly numbered and should be 2416.

Abstract No.	*Line*
5057	7 *for* acetic anhydride *read* active carbon

VOL. 8, 1961

Abstract No.	*Line*
23	2 *for* F. Wolf *read* Friedrich Wolf
54	3 *for* M. G. Allam *read* M. G. E. Allam
134	1 *insert* [**I.**] *after* **cobalt.**
209	3 *for* W. Ulbrich *read* V. Ulbrich
219	2 *for* Skaggs *read* Skeggs
289	2 *for* A. Macek *read* K. Macek
409	24 *for* D. Devoti *read* A. Devoti
449	2 *for* **aesium** *read* **caesium**
490	3 *for* M. J. Gárate *read* M. T. Gárate
543	3 *for* C. H. Bheemasankara Rao *read* C. Bheemasankara Rao
545	3 *for* A. F. Korvezee *read* A. E. Korvezee
562	3 *for* Molnár-Perl *read* Perl
601	4 *for* R. T. Magee *read* R. J. Magee
611	2 *for* K. Burger *read* Kálmán Burger
651	2 *for* A. Milun *read* A. J. Milun
665	2 *for* German Standard *read* Deutscher Normenausschuss
722	2 *for* H. Smith *read* Hylton Smith
724	2 *for* J. Franz *read* Janice Franz
758	2 *for* Berquist *read* Bergquist
779	3 *for* M. Pápay *read* M. K. Pápay
820	4 *for* G. Lyles *read* G. R. Lyles
852	1 *for* L. C. Giddings *read* J. C. Giddings
912	6 *for* 4623 *read* 4632
970	4 *for* Hagai *read* Nagai
981	4 *for* A. K. Bhattacharya *read* Arun K. Bhattacharya
999	3 *for* Tserkovnitsa *read* Tserkovnitskaya
1011	3 *for* M. G. Allam *read* M. G. E. Allam
1037	4 *for* D. K. Banerjea *read* D. Banerjea
1116	1, 2 *interchange* sub-title and main title
1154	1, 2; 8, 9, *for* 3-furaldehyde *read* furfuraldehyde
1198	2 *for* Constantinesco *read* Constantinescu
1336	2 *for* L. Halász *read* I. Halász
1349	1 *insert* **VIII.** *after* **standards.**
1364	10 *for* Ca *read* Cu
1383	24 *for* and *read* or
1406	2 *for* M. Mizera *read* S. Mizera
1406	2 *for* Palaush *read* Palouš
1472	2 *for* K. Burger *read* Kálmán Burger
1473	11 *for* cobaltniitrite *read* cobaltinitrite
1475	2 *for* G. L. Lyles *read* G. R. Lyles
1505	3 *for* Chzhan Fan *read* Fan Chang
1531	3 *for* Perl-Molnár *read* Perl
1591	2 *for* R. J. Amaral *read* J. R. Amaral
1598	4 *for* Molnár-Perl *read* Perl
1652	2 *for* F. Wolf *read* Fritz Wolf
1675	3 *for* Guiseppe *read* Giuseppe
1682	4, 5 *for* 4-Methoxyadrenaline, 4-methoxynoradrenaline *read* 3-Methoxyadrenaline, 3-methoxynoradrenaline
1710	2 *for* E. Lewandowski *read* A. Lewandowski

ERRATA

VOL. 8, 1961

Abstract No.	*Line*	
1835	3	*for* Sheyanov *read* Sheyanova
1837	2	*for* Nikolayev *read* Nikolaev
1864	3	*for* Afanas'ieva *read* Afanas'eva
1876	2	*for* F. P. Sheyanova *read* F. R. Sheyanova
1895	4	*for* Peïzulayev *read* Peïzulaev
1946	3	*for* Ven-Tin Chzhan *read* W.-C. Chang
2014	2	*for* K. Burger *read* Kálmán Burger
2020	2	*insert* **II.** *after* **chromatography.**
2069	2	*for* J. J. D. Ludwick *read* J. D. Ludwick
2110	2	*for* J. Billimoria *read* J. D. Billimoria
2128	1	*for* L. S. Hanka *read* L. J. Hanka
2156	2	*for* F. de Francesco *read* F. De Francesco
2159	2	*for* I. Bastianutti *read* J. Bastianutti
2161	2	*for* I. Bastianutti *read* J. Bastianutti
2224	2	*for* H. Szymanski *read* H. A. Szymanski
2303	3	*for* Popokov *read* Popkov
2328	2	*for* A. N. Busev *read* A. I. Busev
2578	4	*for* Molnár-Perl *read* Perl
2584	2	*for* E. Palaček *read* E. Paleček
2741	2	*for* K. Burger *read* Kálmán Burger
2747	6	*should read* described by which radio-elements, in trace amounts,
2757	2	*for* Mackenzie *read* MacKenzie
2822		The last term in the formula after line 18 should read $(1 - e^{-\lambda/v\omega})$
2844	2	*for* T. L. Blalock *read* T. J. Blalock
2854	2	*for* K. Burger *read* Kálmán Burger
2910	3	*for* K. Burger *read* Kálmán Burger
2916	2	*for* Bártfai *read* Bártfay
2916	3	for *Foly.* read *Lapja*
2974	2	*for* H. Wagner *read* Hildebert Wagner
3172	5	*insert* except Fe *after* cations
3242	2	*for* L. P. Alimarin *read* I. P. Alimarin
3321	5	*for* 1960 *read* 1961
3324	2	*for* S. Bose *read* Sameer Bose
3326	3	*for* K. Burger *read* Kálmán Burger
3452	4	*for* H. Wagner *read* Hildebert Wagner
3461	6	*for* 0·2 to 0·6% *read* 0·0004 to 0·0056%
3560		2nd column, line 21 from the bottom, *for* Korodán *read* Korondán
3560		2nd column, line 24 from the bottom, *for* K. Burger *read* Kálmán Burger
3560		3rd column, line 18, *for* Molnár-Perl *read* Perl
3560		3rd column, line 20, *for* Molnár-Perl *read* Perl
3561	28	*for* Kublek *read* Kublik
3562		2nd column, line 17, *for* K. Freidrich, *read* Klaus Friedrich
3610	2	*for* A. Kharnauchoff *read* A. S. Karnaukhov
3627	3	*for* **Rhodamines S** *read* **Rhodamines B**
3627	10	*for* Rhodamine S *read* Rhodamine B
3627	10	*for* Red 11 *read* Violet 10
3627	25	*for* **Rhodamine S** *read* **Rhodamine B**
3678	6	*for* 1 g *read* 1 μg
3689	2	*for* G. Goldstein *read* Gerald Goldstein
3751	4, 21	*for* G. Goldstein *read* Gerald Goldstein
3787	2	*for* K. Burger *read* Kálmán Burger
3805	12	*for* 1·0N-$(NH_4)_2SO_4.Fe_2(SO_4)_3$ *read* 0·1N-$(NH_4)_2SO_4.Fe_2(SO_4)_3$
3815		*The last line should read* The relative error is usually <5% for 0 to 5 p.p.m.
3818	3	*for* G. Franke *read* Gerhard Franke
3869		*Line* 9 *should read* determined with a coeff. of variation of 3% or greater
4070	3	*for* K. Burger *read* Kálmán Burger
4085	2	*for* Minchevski *read* Minczewski
4085	3	*for* Marchenko *read* Marczenko
4094	4	*for* J. Seals *read* J. M. Seals
4178	3	*for* A. K. Bhattacharya *read* Albani K. Bhattacharya
4193	3	*for* G. Goldstein *read* Gerald Goldstein
4195	2	*for* G. Goldstein *read* Gerald Goldstein
4200	8	*delete* 1961, **8,** 605
4251	3	*insert* **I.** *after* **compounds.**
4274	6	*for* A. Martin *read* Alejandro Martin
4305	2	*for* H. Smith *read* Harry Smith
4381	3	*for* L. Lazer *read* L. S. Lazer
4485	8	*for* Diethyltriaminepenta-acetic *read* Diethylenetriaminepenta-acetic
4493	3	*for* Kallman *read* Kallmann
4525	3	*for* V. P. Zaĭtsev *read* V. N. Zaĭtsev
4535	8	*for* >20 μg *read* <20 μg

ERRATA

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Abstract No.	*Line*
4573	3 *for* Shikar *read* Shakir
4575	4 *for* K. Burger *read* Kálmán Burger
4577	2 *for* F. Wolf *read* Friedrich Wolf
4662	4 *for* K. Burger *read* Kálmán Burger
4677	1 *for* H. Jelínek *read* M. Jelínek
4888	3 *for* K. Friedrich *read* Kurt Friedrich
4906	8 *for* Rhodamine 6G and S *read* Rhodamine 6G and B
4906	8 *for* C.I. Basic Red 1 and 11 *read* C. I. Basic Red 1 and C.I. Basic Violet 10
4979	2 *for* **alcoholic** *read* **methanolic**
4987	3 *for* Aranova *read* Arapova
5172	2 *for* H. Smith *read* Harry Smith
5226	2 *for* G. Goldstein *read* Gilbert Goldstein
5300	3 *for* H. Ulfendahl *read* H. R. Ulfendahl

Cross references after abstract 3977, line 5, *for* Cu *read* Ca

ABBREVIATIONS

Certain abbreviations in everyday use are not included in the following list. When any doubt might arise from the use of an abbreviation or symbol the word is printed in full.

alternating current	a.c.
ampere	amp.
Ångström unit	A
anhydrous	anhyd.
approximate, -ly	approx.
aqueous	aq.
atmospher-e, -ic	atm.
boiling-point	b.p.
British thermal unit	B.Th.U.
calorie (large)	kg-cal.
calorie (small)	g-cal.
centimetre	cm.
coefficient	coeff.
Colour Index	C.I.
concentrated	conc.
concentration	concn.
constant	const.
crystalline / crystallised	cryst.
cubic	cu.
current density	c.d.
cycles per second	c/s
density	ρ
density, relative	d or wt. per ml
dilute	dil.
direct current	d.c.
distilled	dist.
electromotive force	e.m.f.
equivalent	equiv.
ethylenediaminetetra-acetic acid	EDTA
gram	g
gram-molecule	mole
half-wave potential	$E_{\frac{1}{2}}$
hour	hr.
hydrogen ion exponent	pH
infra-red	i.r.
insoluble	insol.
international unit	i.u.
kilogram	kg
kilovolt	kV
kilowatt	kW
liquid	liq.
maxim, -um, -a	max.
melting-point	m.p.
microgram	μg (not γ)
microlite	μl
micromole	μmole
micron	μ
milliampere	mA
milli-equivalent	milli-equiv.
milligram	mg
millilitre	ml
millimetre	mm
millimicrogram	mμg
millimolar	mM
millivolt	mV
minute (time)	min.
molar (concentration)	M
molecul-e, -ar	mol.
normal (concentration)	N
optical rotation	α_l
ounce	oz
parts per million	p.p.m.
per cent.	%
per cent. (vol. in vol.)	% (v/v)
per cent. (wt. in vol.)	% (w/v)
per cent. (wt. in wt.)	% (w/w)
potential difference	p.d.
precipitate (as a noun)	ppt.
precipitated	pptd.
precipitating	pptg.
precipitation	pptn.
preparation	prep.
qualitative, -ly	qual.
quantitative, -ly	quant.
recrystallised	recryst.
refractive index	n_λ^t
relative band speed	R_F
relative humidity	r.h.
revolutions per minute	r.p.m.
saponification value	sap. val.
saturated calomel electrode	S.C.E.
second (time)	sec.
soluble	sol.
solution	soln.
specific gravity	sp. gr.
specific rotation	$[\alpha]_\lambda^t$
square centimetre	sq. cm.
standard temp. and pressure	s.t.p.
temperature	temp.
ultra-violet	u.v.
vapour density	v.d.
vapour pressure	v.p.
volt	V
volume	vol.
watt	W
wavelength	λ
weight	wt.

In addition, the following symbols may be used in conjunction with numerical value or in mathematical expressions—

greater than	$>$	less than	$<$
not greater than	$\ngtr$	not less than	$\nless$
is proportional to	$\propto$	of the order of, approximately	$\simeq$

The Principal Pharmacopoeias are denoted by B.P., U.S.P. or D.A.B., together with the identifying roman numeral or year.

Valency states are represented by a superscript roman numeral, *e.g.*, Fe^{II}, Mo^{V}. Substances in the ionic state are represented by Na^+, Fe^{2+}, Fe^{3+}, etc., for cations and by Cl^-, So_4^{2-}, PO_4^{3+}, etc., for anions.

The chemical nomenclature used in the abstracts is that recommended by The Chemical Society ("Handbook for Chemical Society Authors", The Chemical Society, London, 1960), and may differ from that used in the original articles.

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ANALYTICAL ABSTRACTS

1.—GENERAL ANALYTICAL CHEMISTRY

General reviews of progress, reagents and methods of general application.

1. Progress in analytical chemistry. I. P. Alimarin (V.I. Vernadsky Inst., Akad. Nauk SSSR, Moscow). *Chem. Listy*, 1960, **54** (5), 462–466.—Review.

2. Equipment of the organic chemistry laboratory for gas chromatography. W. Simon (Eidgenössische Tech. Hochsch., Zürich, Switzerland). *Chimia*, 1960, **14** (6), 189–201.—This review includes an introduction to the theoretical and instrumental foundations of gas chromatography, an account of the development and qualitative, quantitative and preparative applications, and a discussion of commercial gas-chromatographic equipment. The products of 37 companies are listed. (98 references.)
E. G. Cummins

3. Extraction in analytical chemistry. B. Trémillon (École Supér. de Phys. et de Chim., rue Lhomond, Paris). *Bull. Soc. Chim. France*, 1960, , 1011–1013.—A discussion is presented, with examples, of the analytical uses of extraction of organic compounds and organometallic complexes in H_2O-immiscible solvents, with special reference to (*i*) the behaviour of the organic phase and (*ii*) selective extraction or separation by controlled displacement of equilibria in the aq. phase.
W. J. Baker

4. Toxicological analysis. A. S. Curry (Home Office Forensic Sci. Lab., Harrogate, England). *J. Pharm. Pharmacol.*, 1960, **12** (6), 321–339.—This review, with 120 references, of developments in the last five years. (*Cf, Anal. Abstr.*, 1956, **3**, 1146.)
A. R. Rogers

5. Radio-frequency methods in analytical chemistry. M. F. C. Ladd and W. H. Lee (Battersea Coll. of Technol., London, England). *Talanta*, 1960, **4** (4), 274–291 (in English).—The theory of radio-frequency measurements of analytical importance is discussed, and suitable types of equipment and applications are reviewed. (166 references.)
W. T. Carter

6. The origin of nuclei in precipitation reactions. B. Fischer (Dept. of Chem., Indiana Univ., Bloomington, U.S.A.). *Anal. Chim. Acta*, 1960, **22** 501–508 (in English).—The pretreatment of water used to dissolve $BaCl_2$ is shown to affect the particle size of $BaSO_4$ subsequently pptd. For a 24 hr.-old $BaCl_2$ soln., typical particle sizes in ordinary laboratory distilled water, in the same water standing for 24 hr. in Pyrex glass, in water distilled in an all-Pyrex-glass system and in fresh high conductivity water are, respectively, 16, 45, 56 and 90 μμ. It is suggested that these phenomena are due to nucleation on sites already present in the water, which are fewer with purer water and which can be removed by adsorption. It is shown that the impurities in analytical-grade chemicals are more than sufficient to provide nucleation sites, other possible sources being containers and colloidal impurities.
R. M. S. Hall

7. Effect of form of reagent on particle sizes of precipitates. R. B. Fischer (Dept. of Chem., Indiana Univ., Bloomington, U.S.A.). *Anal. Chim. Acta*, 1960, **22** (6), 508–514 (in English).—The particle size of ppt. formed when one reagent is in solid form is, in general, one-third to one-tenth of the size obtained when both reagents are in soln. This is illustrated by pptn. of the sulphates of Pb, Ca, Ba and Sr, the fluorides of Ca and Pb, nickel dimethylglyoxime and Ag_2CrO_4. Investigation showed that the presence of a recently dissolved inert electrolyte exerts a variable effect on the particle size of the ppt. These effects are attributed to an increase in nucleation sites provided by the solid reagent, and this is further increased when the reagent is ground.
R. M. S. Hall

8. Omega chrome green BLL: a new analytical reagent. A. A. A. El Raheem, A. S. Moustafa and A.-A. Amin (Publ. Health Lab. and Nat. Res. Centre, Cairo, Egypt). *Z. anal. Chem.*, 1960, **175** (1), 19–29 (in English).—Copper, Pb, Th and Al can be titrated with EDTA in acid soln., with Omega chrome green BLL (C.I. Mordant Green 29) (**I**) as indicator. *Procedure for Cu*—To the sample soln. are added a few drops of indicator (0·1 g of **I** in 100 ml of twice-distilled H_2O), 15 ml of twice-distilled H_2O and 3 ml of 0·01 M HNO_3. From a burette is added an excess of 0·01 M EDTA (disodium salt) and the yellow soln. is titrated with 0·01 M $CuSO_4$ until a violet colour appears. *For Pb*—To the soln. containing $\lessapprox$10 mg of Pb per ml are added a few drops of **I** and 3 ml of 0·01 M HNO_3, and the soln. is titrated to a yellow end-point. *For Th*—To the soln. are added 3 ml of buffer (**II**) (pH 3·45) (25 ml of 0·2 M Na acetate and 475 ml of 0·2 M acetic acid), a few drops of **I** and 2 ml of ethanol. The mixture is warmed to 60° and titrated to a yellow end-point. *For Al*—To the acid sample soln. are added an excess of 0·1 M EDTA and $\simeq$2 ml of **II**; the mixture is boiled for $\simeq$10 min., then cooled in ice to 1° to 3°. After addition of 3 ml of ethanol and a few drops of **I** the soln. is titrated with 0·01 M thorium nitrate. **I** is also recommended as a colour reagent for the spectrophotometric determination of traces of Cu in the presence of a 10-fold excess of Ca, Cd, Co, Mg, Mn and Sr; and for the determination of as little as 0·02 p.p.m. of V in the presence of Al, Ca, Cd, Mg, U, Ni and Sr; the interference of Fe^{3+} is avoided by the addition of a few crystals of ascorbic acid. The results are tabulated and the standard deviation and probable errors of the titrimetric determination of these metals are 11 μg and 7 μg for Cu, 20 μg and 17 μg for Th, 14 μg and 9 μg for Pb, and 14 μg and 9 μg for Al.
B. B. Bauminger

9. **Mixed indicators of methyl red with sulphonated phthalocyanines.** T. P. Sastry and S. A. Pratt, III (Chem. Dept., Texas Southern Univ., Houston, U.S.A.). *Z. anal. Chem.*, 1960, **174** (5), 359–364 (in English).—Copper, cobalt and magnesium phthalocyanine-4,4′,4″,4‴-tetrasulphonates were investigated as components of mixed indicators. When used with methyl red all improved the end-point, giving a green to red colour change. A ratio of 2 parts of methyl red to 3 parts of copper phthalocyanine-4,4′,4″,4‴-tetrasulphonate is recommended for all acid - base titrations for which methyl red is normally used. W. T. CARTER

10. **Metallochromic indicators. IX. Indophenol-complexones.** V. Svoboda, L. Dorazil and J. Körbl (Spolana Neratovice, Czechoslovakia). *Coll. Czech. Chem. Commun.*, 1960, **25** (4), 1037–1043.—The preparation and acid - base and metallochromic properties of a new group of indicators are described and their extinction coeff. were measured. With the use of these indicators the following complexometric titrations can be carried out in chloroacetate buffer soln.—Th^{4+} (5 to 100 mg) at pH 3·3 to 3·5; Fe^{3+} (1 to 30 mg) at pH 2·8 at 60°; Bi^{3+} (4 to 40 mg) at pH 2·8; and Sc^{3+} (1 to 40 mg) at pH 3·5. The sharpest colour changes were obtained with the use of 2,6-dibromophenolindo-*o*-cresol-6′-methyl-aminediacetic acid (Indoferron). J. ZÝKA

11. **Complexometric and argentimetric titrations using chemiluminescent indicators.** L. Erdey and I. Buzás (Tech. Univ., Inst. for General Chemistry, Budapest). *Anal. Chim. Acta*, 1960, **22** (6), 524–531 (in English).—The mechanisms of chemiluminescence of lucigenin (5,5′-biacridyl dimethonitrate) and luminol (3-aminophthalhydrazide) are compared, and the use of these compounds as indicators in the complexometric determination of Cu^{II}, Pb and Hg^{II}, and for the standardisation of EDTA soln., is described. Lucigenin can be used as an adsorption indicator for the argentimetric determination of I^-, even in the presence of Br^- and Cl^-; CN^-, SCN^-, $Fe(CN)_6^{3-}$ and CrO_4^{2-} interfere.
R. M. S. HALL

12. **A heterometric study of the 1,10-phenanthroline compounds with platinum, palladium and gold halides and their analytical application.** M. Bobtelsky and M. M. Cohen (Dept. Inorg. and Anal. Chem., Hebrew Univ., Jerusalem). *Anal. Chim. Acta*, 1960, **22** (5), 485–495.—The apparatus and technique have been described before (*Ibid.*, 1959, **20**, 15, 227 and 357). By changing the composition of the test soln., six different final compounds were obtained for each metal at the first maximum optical density points of the curves, and each compound was suitable for the determination of the metal. Full details of the titrations are given in tabular form. Analytical errors based on these titrations were <1%. With papaverine as reagent, the reactions were approx. four times as sensitive, and in almost all cases only one final compound was formed. R. M. S. HALL

2.—INORGANIC ANALYSIS

General, determination of elements (arranged in the order of the Periodic Table), analysis of minerals and inorganic industrial products.

13. **Detection of ultra-micro quantities of inorganic cations.** D. Exley (Boots Pure Drug Co., Nottingham, England). *Photoelect. Spectr. Gr. Bull.*, 1959, (12), 322–327.—Up to a 50-fold increase sensitivity in flame photometry was obtained b burning a 90% acetone solution of the sample i an oxy-hydrogen flame; the addition of 5% of HC ensures that Na^+, K^+, Ca^{2+} and Mg^{2+} are com pletely in solution. The electronics of a Beckma flame photometer were modified to give greatl enhanced sensitivity, and with the combined tech nique it was possible to detect 3×10^{-13} g of Na and 10^{-10} g of Mg^{2+}. This technique was applie to the determination of 1 to 10 mμg of Na^+, K Ca^{2+} and Mg^{2+} in 0·2 to 0·5-μl samples of inner-ea fluids of cats and guinea-pigs. P. T. BEALE

14. **Differential thermal analysis of hydroxides i reducing atmosphere.** W. Lodding and L. Hamme (Bureau of Mineral Res., Rutgers, State Univ., Ne Brunswick, N.J., U.S.A.). *Anal. Chem.*, 1960, 3 (6), 657–662.—A new high temperature - pressur vacuum furnace is used with the heating elemer outside the tube that forms the pressure vesse No water cooling is required. Gas flows throug the sample and the reference material, and the tw gas streams leave separately. This enables ga analysis of the reaction products to be carried ou while differential thermal analysis is in progres Gibbsite [$Al(OH)_3$] can be determined in the pres ence of hydrated iron oxides. Thermograms fo various mixtures of aluminium and iron oxides ar presented and discussed. D. C. ARMSWORTH

15. **Paper chromatography of inorganic ions i nitrate media. II. Separation of selenium, tellu ium and polonium, and of lead-210, bismuth-21 and polonium.** M. C. Levi and J. Danon (Brazilia Centre for Phys. Res., Rio de Janeiro, Brazil *J. Chromatography*, 1960, **3** (6), 584–585.—Develo on Whatman No. 1 paper for 18 hr. at room temp with a mixture (1:1) of n-butanol and n-propano previously shaken with a soln. containing 7 $LiNO_3$ and 2 N HNO_3. R_F values are ^{210}Pb, 0·27 Te, 0·47; ^{210}Bi, 0·62; Se, 0·72; Po, 0·97.
A. R. ROGERS

16. **Thermolysis of sulphides precipitated b sodium sulphide. II. Sulphides of seleniun tellurium, gold, platinum, ruthenium, lead, bismuth cadmium, silver, palladium and indium.** I. K Taimni and S. N. Tandon (Chem. Lab., Univ. o Allahabad, India). *Anal. Chim. Acta*, 1960, 2 (6), 553–557 (in English).—The sulphides wer examined with a thermobalance and the usua methods of drying and weighing were proved to b satisfactory, except for cadmium sulphide, whic gave results 3% high when the ppt. was dried a the previously recommended temp. of 105° to 115 A new factor of 0·75191 is proposed.
R. M. S. HALL

17. **Application of the RCA 1945 gauge to th analysis of hydrogen in metals.** D. E. Swe (General Motors Corp., Michigan, U.S.A.). *Re Sci. Instrum.*, 1960, **31** (6), 659.—The sample de-gassed in a vacuum furnace, and the evolve gas is expanded via gas pipettes into a known vo ume; the partial pressure of hydrogen in this e panded gas is measured with the RCA gauge (J Nelson, *Ibid.*, 1945, **16**, 273), and the initial hydr gen content of the sample can then be computed Oxygen was found to have an effect on the calibr tion curve, but the effect of oil vapours from t pumps is minimised by the use of cold traps.
G. SKIRROW

8. Determination of deuterium in water and anic compounds. Nobuo Tamiya (Medical and ntal Univ., Yushima, Bunkyo-ku, Tokyo, pan). *Anal. Chem.*, 1960, **32** (6), 724–725.—ter is reduced to H by heating in a sealed tube for ır. at 400° with platinum - zinc or platinum - c - asbestos catalysts (prep. described). The H hen examined in a mass spectrograph. Organic npounds are burnt in a combustion train, with tinum foil and silver gauze as catalysts, in the ıal manner for a determination of H, and the ter produced is frozen in a trap, and analysed described above. The apparatus is illustrated. R. E. E.

19. Determination of helium and neon in gases chromatography. E. Zieliński (School of Mining d Metallurgy ,Kraków, Poland). *Chem. Anal., arsaw*, 1960, **5** (2), 297–307.—The application of s chromatography to the analysis of helium - on mixtures was studied. The column efficiency s analysed as a function of the quality of certain lish adsorbents, and an apparatus was con- ucted to permit satisfactory quant. analysis. rmulae are given and the results are tabulated d discussed. The mean error was $\pm 2{\cdot}0\%$. L. Smakowski

20. Separation of the alkali-metal cations by ctro-chromatography in paper. M. M. Tucker- ın and H. H. Strain (Div. of Chem., Argonne tional Lab., Lemont, Ill., U.S.A.). *Anal. Chem.*, 30, **32** (6), 695–698.—The mobilities of Na^+, K^+, $^+$ and Cs^+ were measured in various organic vents with paper as the stabilising medium. od separations were achieved in 3 hr. on Eaton- keman paper, Grade 301, previously washed with HNO_3 and with H_2O; the electrolyte was 0·2 M monium formate and 0·4 M trichloroacetic acid nitromethane; the potential gradient was 15 V cm. A. R. Rogers

21. Paper chromatography in the separation of as. Separation of alkali and alkaline-earth metals. K. Majumdar and B. K. Pal (Jadavpur Univ., lcutta, India). *Z. anal. Chem.*, 1960, **174** (6), 9–431 (in English).—Ascending paper chromato- phy with the solvent systems 2-ethoxyethanol - O - HCl (7:2:1) or acetone - H_2O - HCl (7:2:1) mits the quant. separation of alkaline-earth tals, including Be and Mg; Li, Na and K can be parated, but Rb and Cs travel with K. R_F lues are tabulated. J. P. Stern

22. Analysis of lithium metal and lithium (ionic) xtures. M. Berkenblit and A. Reisman (Watson b., International Business Machines Corp., lumbia Univ., New York, U.S.A.). *Anal. Chem.*, 30, **32** (6), 721–722.—The procedure is based on e controlled hydrolysis of the metal in a stream of catalytic combination of the evolved H with the rier gas, absorption of the H_2O in P_2O_5, and ration of the hydrolysis residue. The process is plied to the determination of Li in mineral-oil persions, and can be used for other alkali metals. D. C. Armsworth

23. The determination of lithium and its separa- n from other alkali metals. E. Blasius and F. olf (Hardenbergstr. 34, Berlin-Charlottenburg, rmany). *Z. anal. Chem.*, 1960, **174** (5), 349–351 German).—Mixtures of LiCl and NaCl are parated by extracting the LiCl with anhyd. oxan in a reflux apparatus fitted with a P_2O_5 guard-tube and purged with dry nitrogen. Metallic sodium and calcium are added to the flask containing the dioxan to ensure the destruction of traces of water. Macro amounts are separated in 3 to 4 hr., micro amounts in $\simeq 24$ hr. W. T. Carter

24. Indirect complexometric determination of potassium. A. de Sousa (Anal. Chem. Lab., Fac. Sci., Lisbon, Portugal). *Anal. Chim. Acta*, 1960, **22** (6), 522–523 (in French).—Potassium is pptd. as perchlorate and this is reduced to the chloride, which is pptd. as AgCl. The ppt. is dissolved in ammoniacal nickel tetracyanide soln., and the liberated Ni^{2+} are titrated complexometrically with EDTA (disodium salt), with murexide as indicator. R. M. S. Hall

25. Radiometric determination of potassium. V. E. Bel'skiĭ and O. K. Fomin. *Zavod. Lab.*, 1960, **26** (6), 707–709.—The number of impulses (of β-radiation from ^{40}K) per min. per 1% of K is not a constant, but depends on the nature of the sample. By use of a number of compounds of K, as pure salts, mixtures and in solution, it is shown experimentally that this number is inversely proportional to the effective value of Z/A, *i.e.*, of $\Sigma(Z/A)_i P_i/100$, where $(Z/A)_i$ is the ratio of the atomic no. to the atomic wt. of the ith element in the sample and P_i is the percentage content. No correction is necessary, however, when the effective values of Z/A for sample and standard are not significantly different. G. S. Smith

26. Spectrographic determination of potassium, calcium and sodium chlorides in fluxes. N. L. Fishkova and L. N. Komlev (Podolsk Secondary Non-Ferrous Metal Works). *Zavod. Lab.*, 1960, **26** (5), 566–567.—A filtered aliquot (50 ml) of a 10% soln. of the flux mixture is treated with 10 ml of conc. HCl, 5 g of NH_4Cl (a stabilising buffer) and 2 ml of 10% $Co(NO_3)_2$ soln. (internal standard) and then diluted to 100 ml. A spark spectrum is excited with the soln. in a fulgurator (Fishkova and Rŷsina, *Ibid.*, 1959, **25**, 1459), and K (5 to 45%), Na (5 to 39%) and Ca (10 to 36%) are determined by use of the line-pairs K I 4044·14 - Co I 3569·38 A, Na I 3302·99 - Co I 3044·00 A, and Ca I 3630·75 - Co I 3569·38 A. G. S. Smith

27. Colorimetric micro-determination of rubidium and caesium. M. Kobrová (Bäderforschungsinst., Prague II). *Naturwissenschaften*, 1960, **47** (12), 278–279 (in German).—The method is based on conversion of rubidium chloroplatinate and caesium chloroplatinate into platinic iodide and measurement of the extinction, at 496 mμ for Cs and K and at 500 mμ for Rb. To the sample soln. add KI and dil. HCl and measure the extinction after 30 min. It is possible to determine fractions of a microgram per ml. B. Heining

28. Analytical uses of chelones. XXVIII. Detection of copper(II) with 1,2-diaminocyclohexanetetra-acetic acid. F. Bermejo Martínez and M. Guerrero Abella (Univ., Santiago de Compostela, Spain). *Z. anal. Chem.*, 1960, **174** (6), 411–413 (in German).—1,2-Diaminocyclohexanetetra-acetic acid (di-sodium salt) gives a bright blue complex with Cu^{2+} in aq. soln. at a pH between 4·5 and 12. In 10 ml of soln. 60 μg of Cu^{2+} is readily detectable and the limit of sensitivity is $\simeq 25$ μg. Interference by Ni^{2+}, Fe^{3+} and VO^{2+} is severe; colourless ions do not interfere. J. P. Stern

29. **Tritium in radiometric titrations: the copper - anthranilic acid system.** G. H. Aylward, J. L. Garnett, J. W. Hayes and S. W. Law (Univ. of New South Wales, Sydney, Australia). *Chem. & Ind.*, 1960, (20), 560–561.—The results of the radiometric and amperometric titrations of Cu (10 mg) with ^{3}H-labelled anthranilic acid in 50 ml of acetate - acetic acid buffer (pH 4·6) indicate that the two end-points are almost identical. The two titrations were made simultaneously on the same sample, the Wilzbach procedure (*J. Amer. Chem. Soc.*, 1957, **79**, 1013) being used for the radiometric titration. The effect of the base-line error is much less than that of the ^{3}H measurement (1 to 2%).

W. J. Baker

30. **Simultaneous determination of dichromate and cupric ions.** B. K. Sadananda Rao and G. S. Laddha (Alagappa Chettiar Coll. of Technol., Univ. of Madras, Guindy, India). *Z. anal. Chem.*, 1960, **174** (5), 324–328 (in English).—Dichromate and copper can be successively titrated with $TiCl_3$, with diphenylamine and $(NH_4)_2SO_4.FeSO_4$ - KSCN, respectively, as indicators. If the $TiCl_3$ soln. contains Fe^{2+} as impurity two standardisations are necessary, since the Fe^{2+} are active in the first titration but not in the second. *Procedure*—To the sample soln. containing $\simeq$100 mg of $Cr_2O_7^{2-}$ and $\simeq$100 mg of Cu^{2+} add H_2SO_4 (1 : 1) (15 to 20 ml) and dilute to 120 ml with water. Add a small amount of solid $NaHCO_3$ and titrate with 0·1 N $TiCl_3$, adding diphenylamine (0·005 M in conc. H_2SO_4) (0·2 ml) just before the end-point. Then add 10% KSCN soln. (10 ml), a few drops of 0·1 N $(NH_4)_2SO_4.FeSO_4$ and some solid $NaHCO_3$ and titrate with 0·1 N $TiCl_3$ until the colour changes from red to green. For 100-mg quantities of Cu^{2+} and $Cr_2O_7^{2-}$ the error is $\simeq \pm 0{\cdot}5\%$.

W. T. Carter

31. **Controlled-potential electrolytic determination of copper in lead and tin-base alloys.** B. Alfonsi (Res. and Control Lab., Auto-Avio, Fiat, Torino, Italy). *Anal. Chim. Acta*, 1960, **22** (5), 431–436.—Lingane's methods ("Electroanalytical Chemistry," Interscience Publishers, N.Y., 1953) have been extended for use with 5-g samples. For lead alloys, Ag and Bi are co-deposited with Cu and must be determined separately, and with concn. of Sb > 1·5% the deposit must be dissolved and re-electrolysed to avoid errors due to the co-deposition of Sb. Iron inhibits the deposition of Sb, but interferes with the determination of Cu. For tin alloys also, Ag and Bi are co-deposited with Cu; Pb and Cu can be determined successively. For lead alloys the accuracy is within 3% (12 determinations), and for tin alloys within 2% (15 determinations). Full instructions for the dissolution of the samples and the determinations are given.

R. M. S. Hall

32. **Analysis of copper-refinery electrolyte by a condensed d.c. arc solution technique.** N. Tomingas (Canadian Copper Refiners Ltd., Montreal East, Quebec, Canada). *Appl. Spectroscopy*, 1960, **14** (3), 72–75.—One drop of the sample is loaded on to the flat end of a graphite electrode (0·25 in. in diam.) and two such electrodes are arced together, with a Hilger-BNF type source unit at 210 μF, 15 ohms and 0·03 mH. Electrodes are rendered uniformly porous by being heated for 1 hr. at 540°. Cobalt chloride is added to the sample solutions as an internal standard for the determination of As, Sb, Ni, Fe and Bi in the range 0·1 to 10 g per litre with a coeff. of variation of $\simeq$3%.

P. T. Beale

33. **Spectrographic analysis of gold for purity.** O. F. Degtyareva and M. F. Ostrovskaya. *Zavod. Lab.*, 1960, **26** (5), 564–566.—The flat end of a carbon rod is treated with a drop of aqua regia and ignited in a d.c. arc (20 to 30 amp.) for 30 to 45 sec. to remove Cu, Al and Fe. It is then coated with polystyrene lacquer (3·3% in benzene) and dried for 1 min. in air. A portion (0·04 ml) of the soln. of the sample prepared by dissolution in aqua regia and addition of an equal vol. of water, or of a standard soln. (containing 10% of Au), is placed on the treated end of the rod, which is then heated first at 80° to 90° and then at 160° to 170° to evaporate the soln. The spectrum is excited in an a.c. arc (6 amp.) and the elements Be, Cu, Mn, Ca, Mg, Cd, Al, Fe, Cr, Ba, Ni, K, Co, Pb, Zn and Ag are determined.

G. S. Smith

34. **Polarographic analysis of gold and gold alloy plating solutions.** A. E. Knotowicz and G. Tatoian (The Patent Button Co., Waterbury, Conn., U.S.A.). *Plating*, 1960, **47** (6), 645–647.—The determination of Au, and of Ag, Cu, Ni and Zn as impurities or as components of cyanide plating soln., is described. For the determination of Ag and Au, use N KCN as supporting electrolyte, and read the diffusion current for Ag at −0·95 V and for Au between −0·95 and −1·7 V. Nickel gives a similar wave to Au so that a correction must be applied if Ni is present. For the determination of Cu, Ni and Zn, use N aq. NH_3 - N $(NH_4)_2SO_4$ as supporting electrolyte. Add 1 ml of conc. HNO_3 and 3 ml of conc. H_2SO_4 to the test soln. and heat to fuming. Cool, add gelatin soln. and dilute to 50 ml with the electrolyte, allowing the ppt. of Au to settle. Read the current for Cu at −0·8 V (correct for Ag), for Ni between −0·8 and −1·275 V, and for Zn between −1·275 and −1·6 V. Results for Au, Ag, Cu and Ni differ from chemical values by about 1%, for Zn by about 5%.

P. D. Parr-Richard

35. **Rapid determination of gold in ores and rocks for prospecting purposes.** V. Patrovský (Inst. Mineral Ores, Prague). *Geol. Průzkum*, 1959, (10), 302–303.—*Procedure*—To the finely powdered sample (0·5 to 10 g, containing 0·002 to 0·05% of Au) add conc. HCl (5 to 25 ml) and conc. HNO_3 (1 to 5 ml) and heat slowly on a water bath. Evaporate to a syrup and add HCl (1 ml) and H_2O (30 ml). Filter and wash the ppt. with H_2O (> 20 ml). To the filtrate add KBr (3 to 5 g) and extract with 15 ml and 10 ml of ethyl ether. Evaporate the combined extracts to dryness and treat the residue with HCl (1 ml) and bromine water (2 ml). Remove the Br by boiling, evaporate to dryness on a water bath and dissolve the residue in HCl (1 : 1) (0·5 ml). Dilute with H_2O (30 ml), add a satd. aq. soln. of salicylic acid (0·5 ml), conc. H_3PO_4 (0·2 ml) and 1% benzidine soln. in 20% acetic acid (1 ml), dilute with H_2O to 50 ml and measure the extinction with a violet filter. Compare with a calibration curve. Five samples can be analysed within 1·5 to 2 hr.

J. Zýka

36. **The cupellation of gold - aluminium alloys.** A. M. Scherhaufer (Hauptpunzierungs und Probieramt, Wien, Austria). *Z. anal. Chem.*, 1960, **174** (5), 352–354 (in German).—Gold - aluminium alloys containing 75% of Au which cannot be subjected to direct cupellation can be analysed by dissolvin

sample in 5 N NaOH, filtering off the residual d and carrying out a normal cupellation. The thod is considerably more rapid than the chem- l separation of Au with H_2SO_3, and gives results eeing to within 0·1%. W. T. CARTER

37. **[Spectrographic] determination of beryllium tal in air.** R. J. Powell, P. J. Phennah and J. E. ll (Res. Lab., General Electric Co. Ltd., Wembley, gland). *Analyst*, 1960, **85**, 347–353.—The sample btained by mechanical aspiration of air through er-paper. The paper is wet-ashed with H_2SO_4 l a Br - HNO_3 mixture or, more rapidly, with a xture of $HClO_4$ (0·5 ml), H_2SO_4 (1 ml) and Br - NO_3 (5 ml). The residue is diluted, $Ca_3(PO_4)_2$ n. is added followed by drops of aq. NH_3 until tn. is complete. The ppt. is separated and washed centrifuging, then dissolved in 0·2 ml of a soln. $AuCl_3$ in HCl, the Au line 2913 A serving as an ernal standard. The vol. of the soln. is adjusted 1 ml and an aliquot (0·1 ml) is sparked in a ous graphite cup. The intensity of the Be line 30 A) is measured, corrected for background and erred to a working graph prepared from stand- s ranging from 0 to 50 μg of Be per ml by the ne analytical procedure. The method has been d only for samples consisting of metallic beryl- n and the oxide produced by atmospheric oxida- n. Modification of the chemical treatment might necessary with beryllia refractories and other npounds of Be. A. O. JONES

38. **Spectrophotometric determination of beryllium air and dust.** R. H. A. Crawley (English Electric Ltd., Atomic Power Division, Whetstone, gland). *Anal. Chim. Acta*, 1960, **22** (5), 413–420. Membrane air filters, types AF400 and AF600, ve been shown to be much more efficient in pping beryllium dust than No. 41 filter-papers, l ignition of the filter is also quicker. The ashed er is fused with $KHSO_4$, the melt is extracted h dil. HCl, and any silica is removed by centri- ing. EDTA (disodium salt) is added to mask erference from <2 mg of Ca, Mg, Zn, Al, Ti, Co, V and Ni, and nitrilotriacetic acid to eliminate erference from Zr and reduce that due to Cr. e pH of the soln. is adjusted to between 4 and 7 h dil. aq. NH_3, a buffered soln. of aluminon H 5·8) is added, and, after making up the soln. an appropriate vol. and heating on a water bath complete the colour development, the extinction measured at 530 mμ. If Fe is present, a cor- tion must be made. Results are referred to a bration graph covering the range 0 to 8 μg of The lower limit of detection is 0·1 μg of Be, l recoveries on 0·5 to 5·0 μg range from 90 to %. R. M. S. HALL

39. **The use of hexa-amminocobalt chloride elled with cobalt-60 for radiometric determination. I. Determination of beryllium and bismuth.** Dema, I. Gainar and T. Nascuţiu. *Rev. Chim., charest*, 1960, **11** (5), 291–293.—The sensitivity the method of Pirtea for Be (*Anal. Abstr.*, 7, **4**, 2900) has been increased to 5 μg. For Bi method of Pop (*Acad. R.P.R., Stud. Cercet. m.*, 1954, **2**, 245), based on the pptn. of $Cl_6][Co(NH_3)_6]$, is used. *Standardisation of gent*—A 2% soln. of $[^{60}Co(NH_3)_6]Cl_3$ (**I**) (0·1 ml, vity $\simeq 10^4$ counts per mg of Co) is placed on lter-paper disc, superimposed on an aluminium , of the same thickness in a special Plexiglas r-crucible (illustrated). The paper is dried lamp) and its activity measured in the above units in a G. - M. counter. *Determination of Be*—The faintly acid test soln. (1 ml ≡ 0·005 to 0·5 mg of Be) is treated with a slight excess of solid ammonium carbonate until the ppt. at first formed re-dissolves. The soln. of **I** (0·5 to 1 ml in excess) is then added dropwise, with stirring and rubbing the sides of the beaker, and the mixture is set aside for 15 min. The ppt. is then filtered off in the special crucible, washed with 60% ethanol containing a few drops of a conc. soln. of inactive **I**, then washed with 96% ethanol. The crucible is washed externally with the 60% ethanol soln. to remove traces of active material, wiped dry with filter-paper, then dried, and the activity (counts per min.) measured as described above, with the same counter and geometrical conditions. Then Be (mg) = 0·3060 × activity of ppt./activity of **I** soln. The maximum error for the range 0·007 to 0·35 mg of Be is −14·3%. *Determination of Bi*—The test soln. in 0·5 N HCl (1 ml ≡ 0·01 to 0·5 mg of Bi) is treated with **I** soln. as described above, and set aside for 1 to 2 hr. (overnight for 0·01 to 0·05 mg of Bi). The ppt. is then filtered off, washed with a soln. containing 2·5 ml of conc. HCl and 0·05 g of inactive **I** per 100 ml, and treated as described above; Bi (mg) = 3·546 × activity of ppt./activity of **I** soln. The maximum error for the range 0·008 to 0·394 mg of Bi is −25%. R. E. E.

40. **Spectrographic determination of calcium and magnesium fluorides in aluminium electrolytic baths.** I. L. Reznikov (Aluminium Works, Nadvoitsk). *Zavod. Lab.*, 1960, **26** (5), 570–574.—The soln. is placed in the hollow of an aluminium electrode and sparked with a flat-ended counter-electrode. Samples and standards should contain similar concn. of cryolite. The intensities of the lines Mg II 2798·06, Mg II 2790·79 and Ca II 3179·33 A are compared with Na I 3302·33/99 A. The method is applicable to contents of CaF_2 and MgF_2 from 1·5 to 10%. Carbon (>2%) interferes.

G. S. SMITH

41. **Colorimetric micro-methods for the determination of calcium based on the formation of coloured azo-derivatives of 8-hydroxyquinoline.** V. Armeanu and P. Costinescu. *Rev. Chim., Bucharest*, 1960, **11** (6), 343–345.—The methods are based on the reaction of the Ca - 8-hydroxyquinoline complex with diazotised sulphanilic acid (**I**) or 7-amino-1-naphthol-3-sulphonic acid (**II**). *Separation of Ca*—The neutral test soln. (10 μg of Ca per ml) (5 to 10 ml) is warmed to 60°, treated with 2 to 5 ml of 2 N aq. NH_3 and 1 to 2 ml of 0·01 M ethanolic 8-hydroxyquinoline and heated gently till the ppt. becomes crystalline. The mixture is cooled to 40° to 50°; the ppt. is filtered off, washed with water till free from 8-hydroxyquinoline, then re-dissolved in 2 to 3 ml of warm 2 N HCl and diluted to 50 ml (soln. *A*). *Procedure with* **I**—The reagent is prepared by dissolving 0·1 g of **I** in 50 ml of ethanol, adding 10 ml of conc. HCl and diluting to 100 ml with water; to 20 ml is added 1 to 2 ml of 0·1 M $NaNO_2$ and the mixture is diluted to 100 ml. An aliquot (5 to 30 ml) of soln. *A* is treated with 5 ml of reagent followed by 5 ml of aq. NH_3 (1 : 4) and diluted to 50 ml with water. The extinction is read at once or after 24 hr. with or without a 480-mμ filter. The sensitivity is 1 in 4×10^7 and the coeff. of variation is 0·09% with, and 0·11% without, the filter. *Procedure with* **II**—The reagent is prepared by dissolving 0·2392 g of **II** in 2 N Na_2CO_3 and diluting to 100 ml; 20 ml is treated with 3 ml of 0·1 M $NaNO_2$ and 2 ml of 2 N HCl and diluted to 100 ml.

An aliquot (5 to 40 ml) of soln. *A* is treated with 5 ml of reagent and 5 ml of NaOH soln. and diluted to 50 ml. The extinction is read at once or after 24 hr., with or without a 430-mμ filter. The sensitivity is 1 in $2{\cdot}3 \times 10^7$ and the coeff. of variation is 0·11% with, and 0·12% without, the filter.

R. E. E.

42. **Micro-titration of calcium with EDTA. Magnesium interference.** J. C. van Schouwenburg (Lab. of Soils and Fertilisers, Agric. State Univ., Wageningen, Netherlands). *Anal. Chem.*, 1960, **32** (6), 709–711.—The low results caused by Mg in the EDTA titration of Ca with murexide is probably due to adsorption of the Ca - murexide complex on the pptd. $Mg(OH)_2$. This is eliminated by the addition of Carbocel (Na carboxymethylcellulose) (**I**). *Procedure*—To the neutral test soln. (1 to 6 ml) add 25% (v/v) aq. triethanolamine soln. (0·2 ml), 3·2% aq. KCN soln. [containing 3 ml of 10^{-3} M EDTA (**II**) per 100 ml] (2 drops), 0·5% aq. **I** soln. (containing 5 ml of **II** per 100 ml) (0·2 ml) and N NaOH (0·4 ml), stirring after each addition. Set aside for 30 min., then add fresh 0·025% aq. murexide (Merck) soln. (0·1 ml), dilute the mixture to 8 ml and titrate with **II**. Phosphate does not interfere; Mg causes a slight positive error, and Zn and Mn may interfere if the Ca is not in comparatively large excess. R. E. E.

43. **Complexometric determination of calcium in calcium molybdate.** N. M. Rakhmilevich. *Zavod. Lab.*, 1960, **26** (6), 717.—The sample (0·5 g) is dissolved in 25 ml of conc. HCl, an aliquot (50 ml) of the diluted soln. (250 ml) is diluted to 150 ml, 2 to 3 drops of a soln. prepared by dissolving 0·25 g of indigo carmine in 100 ml of 25% ethanol are added, and the soln. is treated with 20% NaOH soln. until a yellow colour appears. It is then titrated with 0·05 M EDTA, in the presence of 0·1 to 0·15 g of murexide, to the appearance of a violet colour. G. S. Smith

44. **Complexometric titration of calcium in the presence of large amounts of aluminium [in fluorspar and other minerals].** M. A. Popov (Central Lab., Geol. Dept., Novosibirsk). *Zavod. Lab.*, 1960, **26** (5), 540–542.—The sample (0·25 g), after treatment with 8 ml of 10% acetic acid soln. followed by washing with water to remove CO_3^{2-}, is dissolved in 20 ml of neutral 8% $AlCl_3$ soln. The filtered soln. is diluted to between 120 and 150 ml and treated with either 15 ml of water - glycerol (1 : 1) or 10 ml of 15% sucrose soln. After addition of 2 or 3 drops of 0·2% indigo carmine soln., the soln. is made alkaline with 20% NaOH soln. (3 to 5 ml in excess) and treated with 1 to 3 drops of 10% Na_2SO_3 soln. and 5 to 7 drops of Acid chrome dark blue (C.I. Mordant Blue 7) indicator soln., prepared by dissolving 0·5 g in 25 ml of aq. NH_3 (sp. gr. 0·91) and diluting with ethanol to 125 ml. The Ca is then titrated with 0·04 M EDTA (disodium salt). G. S. Smith

45. **Flame-photometric determination of strontium in silicates.** M. Fornaseri and L. Grandi (Ist. di Geochim., Univ. Roma, Italy). *Geochim. et Cosmoch. Acta*, 1960, **19** (3), 218–221.—The sample (1 g) is decomposed with $HClO_4$ (5 ml) and HF (20 ml), then evaporated to dryness, and the residue is dissolved in hot water; Ca soln. (1%) (10 ml) is added to compensate for the effect of Al in silicates low in Ca, the soln. is made up to 50 ml, and divided into two 25-ml aliquots. The first is diluted to 50 ml to give soln. *A*; the second is mixed with a soln. of Sr (25 p.p.m. of SrO) (10 ml) and made up to 50 ml to give soln. *B*; *A* and *B* contain, respectively, x and $(x + 5)$ p.p.m. of SrO. The emissions are then measured at 461 mμ and corrected by deducting the mean of the emissions at 454 and 463 mμ. Then $x = 5E_1/(E_2 - E_1)$, where E_1 and E_2 are the corrected emissions for *A* and *B*, respectively. The coeff. of variation ranged from 2 to 3·25% for the range 100 to 1500 p.p.m. of Sr.

R. E. E.

46. **Gravimetric determination of strontium oxide in Portland cement.** C. L. Ford (A.S.T.M., 1916 Race Street, Philadelphia, Pa., U.S.A.). *Bull. A.S.T.M.*, 1960, (245), 71–75.—A comparative study of four different methods of determining small amounts of acid-sol. strontium in cement shows that the most suitable and accurate gravimetric procedure is that based on extraction of $Ca(NO_3)_2$ into 80% HNO_3 at 80° to 82° from the dry mixed nitrates of Ca and Sr. The residual insol. $Sr(NO_3)_2$ is washed several times with 80% HNO_3 and dried to const. wt. at 130° to 140° for ≃2 hr. The SiO_2, mixed oxides and Mn are removed initially by standard procedures, and the ignited ppt. of the mixed oxalates (one pptn. only is made) is used for the separation of Ca and Sr. The rate of heating (6 to 8 min.), temp. and very rapid stirring during the first 5 min. are critical for a successful separation; stirring time should be from 40 to 60 min according to the approx. content of SrO. The lower limit of determination is ≃0·05% of SrO and the values are usually about 0·02% lower than those obtained by flame photometry.

W. J. Baker

47. **Complexometric determination of cation mixtures. II. Complexometric titration of zinc and copper in the presence of murexide as indicator.** C. Liteanu, G. Murgu and L. Marinescu (Univ. "Babes-Bolyai," Cluj, Romania). *Z. anal. Chem.*, 1960, **175** (1), 1–4 (in German).—To determine the sum of Cu and Zn, the sample containing ≃60 mg of Cu^{2+} and Zn^{2+} is adjusted to pH ≃5 with NaOH, 10 ml of M NH_4Cl is added and the ppt. is dissolved in aq. NH_3. An aliquot of this soln. (*A*) at pH 8 to 9 is titrated at room temp. with 10^{-2} M EDTA in the presence of indicator (1 part of murexide and 200 parts of NaCl) to a colour change from yellow to violet. Zinc is determined in another aliquot of soln. *A*. After dropwise addition of M KCN until the aliquot is decolorised, and then 1 ml in excess, 2% formaldehyde soln. is added, and the pptd. $Zn(OH)_2$ is dissolved in aq. NH_3. The pH is adjusted to 8 to 9, and the soln. is titrated with 10^{-2} M EDTA, with murexide as indicator, at room temp. to a colour change from yellow to violet. The method is applicable to the analysis of ores, alloys and electroplating soln. B. B. Bauminger

48. **Analysis of cyanide zinc baths. Rapid determination of metal and total cyanide.** A. Salk (Nymanbolagen AB, Uppsala, Sweden). *Metal Finish.*, 1960, **58** (5), 59–60.—Dilute 10 ml of the soln. to 100 ml. To 10 ml of the diluted soln. add H_2O (100 ml), 20% NaOH soln. (5 ml) and dipheny carbazide indicator. Titrate with 0·1 N $AgNO_3$ until the red colour changes to yellow; hence calculate the NaCN. Add N NH_4Cl (10 ml) and conc. aq. NH soln. (5 ml) and adjust the pH to 8·0 to 8·5 with dil HCl. Titrate with 0·1 M EDTA (disodium salt to Eriochrome black T; hence calculate the Zn.

N. E.

49. **Determination of small amounts of zinc in tallic cadmium.** P. V. Marchenko (Inst. of Gen. d Inorg. Chem., Acad. Sci., UkrSSR). *Zavod. b.*, 1960, **26** (5), 532–535.—The sample (2 to) is dissolved in 20 to 50 ml of 4 N HCl and the n. is evaporated to dryness. The residue is solved in water and the soln. (50 ml) in a separng-funnel is treated first with 10 ml of 4 N HCl d 18 ml of H_2SO_4 (1:1) and then gradually with ml of methylene blue soln. (prepared by treating) ml of a 1% soln. with 1 ml of conc. H_2SO_4 d 2 ml of 4% NH_4SCN soln. and filtering after to 2 days) and 20 ml of 7·6% NH_4SCN soln. e soln. is diluted to 100 ml and set aside for 30 40 min. and then shaken with 5 ml of toluene so at the ppt. appears interfacially. The aq. layer rejected and the ppt. is washed with 1% NH_4SCN n. (3 × 10 ml), then dissolved in 10 ml of 4 N Cl and re-pptd. without removal from the separng-funnel. The washed ppt. is dissolved in nl of H_2SO_4 (1:3), 1 ml of 30% H_2O_2 is added, d the soln. is evaporated to fuming. From the ghtly acid soln. Cu is removed by extraction with hizone soln. and the soln. at pH 5·5 is treated th 10 ml of 50% $Na_2S_2O_3$ soln. followed by traction of the Zn with dithizone soln. The tract is washed first with a soln. prepared by xing 75 ml of 20% Na acetate soln. and 10 ml of % $Na_2S_2O_3$ soln., diluting to 460 ml and adding ml of 10% HNO_3 soln., then with 50% $Na_2S_2O_3$ n., and finally with water. The Zn is re-extracted th HCl (1:1000) and the soln. is treated with 10 of 20% Na acetate soln. The Zn is then extracted ain with dithizone soln., the extract is washed th 0·04% Na_2SO_3 soln., and the extinction is asured. The method is sensitive to 0·5 μg of in 5 ml of extract. G. S. Smith

50. **Colorimetric determination of zinc and cadium with 8-quinolinol.** W. L. Medlin (Field Res. b., Magnolia Petroleum Co., Dallas, Tex., S.A.). *Anal. Chem.*, 1960, **32** (6), 632–634.—*termination of Zn*—The sample (≯100 μg of Zn) issolved in 7% HCl and the pH is adjusted to 8·8 9·5 with aq. NH_3; the reagent (5 g of 8-hydroxynoline in 100 ml of H_2O containing 15 ml of cial acetic acid) is added and the yellow complex extracted with $CHCl_3$ and measured spectrootometrically at 400 mμ. The method is valuefor the determination of Zn in the presence of ge amounts of Ca; for other applications it is ch less sensitive than the zincon method. *Deterzation of Cd*—The same procedure is followed cept that the pH is adjusted to 7·6 to 8·6. The thod offers no particular advantages over existing cedures, as the presence of Ca is not a serious blem in the determination of Cd.

R. E. E.

51. **New indirect volumetric method for the determination of cadmium.** J. Goldstein. *Rev. Chim., Bucharest*, 1960, **11** (6), 334–336.—The proposed thod is based on the pptn. of $CdK_2[Fe(CN_6)]$ the pptn. of the excess of $K_4Fe(CN)_6$ (**II**) by a ndard soln. of $AgNO_3$ and back-titration of the ess of $AgNO_3$ with a soln. of NH_4SCN, with $H_4)_2SO_4.Fe_2(SO_4)_3$ as indicator. *Procedure*—A . of the sample in H_2SO_4 neutralised to methyl nge with 10% aq. NH_3 is treated with 15 to 25 g $NH_4)_2SO_4$ (6 to 10 g per 100 ml of soln.) and an ess of a standard soln. of **II**. The mixture, tted to 250 ml, is stirred, the pptd. **I** is allowed settle for a few minutes, and then filtered off a dry paper. A 50-ml aliquot of the filtrate is treated with an excess of 0·1 M $AgNO_3$. The pptd. $Ag_4Fe(CN)_6$ is filtered off immediately and a 50-ml aliquot of the filtrate is titrated with 0·1 M NH_4SCN. The method can be used in the presence of the alkali and alkaline-earth metals, but ions that are pptd. by either $[Fe(CN)_6]^{4-}$ or Ag^+ interfere. The error is ≯±0·1% on the macro scale and ±0·5% on the micro scale. H. Sher

52. **Gravimetric determination of mercury by precipitation with sodium tetraphenylborate.** A. Heyrovský (Charles' Univ., Prague, Czechoslovakia). *Analyst*, 1960, **85**, 432–434.—To the nearly neutral or faintly acid sample soln. (5 to 100 mg of Hg^{2+} at a concn. of ≃1 mg per ml) are added a few ml of satd. Na acetate soln. and then an excess of aq. Na tetraphenylborate soln., drop by drop, with stirring. The ppt. is collected in a sintered glass crucible, washed with a satd. aq. soln. of diphenylmercury, dried in a desiccator and weighed. Cations that form insol. ppt. with Na tetraphenylborate (*e.g.*, Ag^+, Tl^+, K^+, Cs^+, Rb^+ and NH_4^+), anions that form insol. phenylmercury salts (*e.g.*, Cl^-, Br^-, I^- and SCN^-) and Hg^+ interfere. Interference by metal ions pptd. as hydroxide or basic salt at the pH used can be avoided by addition of a chelating agent (*e.g.*, EDTA). Quoted results indicate an accuracy within 1%. A. O. Jones

53. **Titration of mercury(II) with sodium tetraphenylborate.** A. Heyrovský (Charles' Univ., Prague, Czechoslovakia). *Anal. Chim. Acta*, 1960, **22** (5), 405–408.—The titration reaction proceeds in two stages—(*i*) 0·25 mole of Na tetraphenylborate (**I**) reacts with 1 mole of Hg^{2+} to form a clear soln. of phenylmercury salt; then (*ii*) further addition of **I** causes pptn. of diphenylmercury, and a persistent turbidity is taken as the end-point. For titration, the sample soln. should contain 5 to 50 mg of Hg^{2+}, and not more than 1 mg per ml. The soln. can be neutral, or slightly acid (0·05 N in HNO_3 or $HClO_4$) or buffered with Na acetate. The use of EDTA in buffered soln. eliminates interference from Cu, Zn, Mg, Ca, Mn and Ni; Ba, Fe, Co, Pb, Al and Bi do not interfere in acid soln.; and there is no interference from NO_3^-, ClO_4^-, PO_4^{3-}, ClO_3^-, SO_4^{2-}, NO_2^- or AsO_4^{3-}. Any anion that gives an insoluble phenylmercury salt (Cl^-, Br^-, I^- or SCN^-) interferes, as do Hg^+, but K^+ in concn. <3 times that of Hg^{2+} can be tolerated. Silver interferes in concn. >0·5 that of Hg^{2+}. When tested with $Hg(NO_3)_2$ and $Hg(ClO_4)_2$, the method had an error of <1% (for 2 to 30 mg of Hg^{2+}). R. M. S. Hall

54. **Potentiometric determination of mercury(II) with ethylenediaminetetra-acetic acid.** H. Khalifa and M. G. Allam (Anal. Chem. Dept., Cairo Univ., Giza, Egypt). *Anal. Chim. Acta*, 1960, **22** (5), 421–427.—Mercury (200 μg to 100 mg) alone is determined with EDTA (disodium salt) (0·05 to 0·005 M) at pH 8 to 10, with a silver amalgam *vs.* S.C.E. system for the potentiometric end-point. The optimum pH for 0·005 M soln. is 6; 200 μg of Hg^{2+} can then be determined. Aluminium, Fe, Ti, Be and the alkaline-earth metals can be complexed or pptd. by F^-, which do not interfere. With binary mixtures of Hg and Ba, back-titration of excess of EDTA at pH 8 can be used to determine Hg, and at pH 11 for the determination of both metals. With binary mixtures of Hg and Pb, Mn or Zn, the total metal content can be determined as described above, and the titration repeated with Hg masked by I^-. With binary mixtures of Hg^{2+} and Ni, Cd or Co, the Hg^{2+} are titrated with KI

soln. and the total metals are determined by back-titration with $Hg(NO_3)_2$. Results are accurate to within 0·5%. R. M. S. Hall

55. **Catalysts for cerium(IV) oxidimetry. Determination of mixtures of mercury(I) and mercury(II).** W. H. McCurdy, jun., and G. G. Guilbault (Univ. of Delaware, Newark, U.S.A.). *Anal. Chem.*, 1960, **32** (6), 647–650.—A mixture of $AgNO_3$ and $Mn(NO_3)_2$ in $HClO_4$ (prep. described) is used as a catalyst in the oxidation of Hg^I to Hg^{II} with $Ce(SO_4)_2$. The test soln. (15 to 300 mg of Hg^I as perchlorate, nitrate or sulphate) is treated with a 50 to 60% excess of $Ce(SO_4)_2$ soln. (prep. described) and 1 ml of catalyst, and sufficient $HClO_4$ to make the soln. 2 N. The mixture is heated at 90° till the colour becomes cherry-red (3 to 5 min.) and then cooled rapidly in ice, and the excess of $Ce(SO_4)_2$ is back-titrated with $(NH_4)_2SO_4.FeSO_4$ soln., with ferroin as indicator; 10 to 600 mg of Hg^I can be determined in the presence of 1 g of Hg^{II} with a coeff. of variation of ±0·3%. Bivalent mercury in $HClO_4$ soln. can be reduced to Hg^I by shaking with metallic mercury, and then determined as described above. Interferences are discussed. R. E. E.

56. **Colorimetric estimation of aluminium with catechol violet.** A. Anton (Textile Fibres Dept., E. I. du Pont de Nemours and Co., Inc., Wilmington, Del., U.S.A.). *Anal. Chem.*, 1960, **32** (6), 725–726.—Dissolve 0·3864 g of catechol violet in H_2O (100 ml). Mix 77 ml of reagent-grade pyridine with glacial acetic acid (63 ml) to give a buffer of pH 5. *Procedure*—To the test soln. (≯1·5 mg of Al) add buffer (10 ml) and reagent soln. (2 ml). Dilute to 100 ml with H_2O and measure the extinction against a reagent blank at 615 mμ. Zinc, Mg and Mn do not interfere up to 50 p.p.m., but Fe, Cu, Bi and Sn in concn. >2 p.p.m. interfere.
D. C. Armsworth

57. **Complexometric determination of aluminium and lead ions, with variamine blue as indicator.** L. Erdey and L. Pólos (Inst. of General Chem., Tech. Univ., Budapest, Hungary). *Z. anal. Chem.*, 1960, **174** (5), 333–337 (in German).—An indirect method is described in which the indicator system variamine blue, $K_4Fe(CN)_6$ and $K_3Fe(CN)_6$ at pH 5 is used (*cf. Anal. Abstr.*, 1958, **5**, 2547). *Procedure*—To the sample soln., containing 6 to 60 mg of Al or 20 to 500 mg of Pb, add 20 to 100 ml of 0·05 M EDTA (disodium salt) and 10 ml of buffer soln. (95·93 g of Na acetate and 295 ml of M acetic acid, made up to 1 litre with water). Boil for 3 to 4 min., then cool. Add 0·2 g of indicator [50 g of NaCl, 0·2 g of variamine blue, 0·422 g of $K_4Fe(CN)_6.3H_2O$ and 0·082 g of $K_3Fe(CN)_6$] and titrate with 0·05 M $ZnSO_4$. The alkali and alkaline-earth metals do not interfere. The standard deviations for 11 determinations on 27 mg of Al and 207 mg of Pb were ±0·065% and ±0·165%, respectively. W. T. Carter

58. **Flame-photometric determination of small quantities of sodium in metallic aluminium.** K. H. Neeb (Forschungslab. Siemens Schuckertwerke A.-G., Erlangen, Germany). *Z. anal. Chem.*, 1960, **174** (5), 328–333 (in German).—Concn. of Na in aluminium below 0·001% are difficult to determine by flame photometry because of the increasing background from Al and the negative effect of Al on the emission of Na. Most of the Al can be removed simply and without contamination by reaction with ethyl bromide in a quartz apparatus to form $Al_2(C_2H_5Br)_3$, which is distilled off *in vacuo*. The residue is dissolved in dil. HCl and analysed by flame photometry. For a 2-g sample the lower limit of detection is 0·00001% of Na and the relative error is ±10%. In the range 0·0001 to 0·001% the relative error is ±5%. Calcium can be determined in a similar manner with a detection limit of 0·0005% and a relative error of ±10%.
W. T. Carter

59. **Quantitative spectrographic determination of gallium in solution.** A. Újhidy. *Magyar Kém. Foly.*, 1960, **66** (4), 132–134.—The vibrating a.c. arc method of Schiebe and Rivas (*Angew. Chem.*, 1936, **49**, 443) and the rotating graphite disc method of Boyle *et al.* (*J. Lab. Clin. Med.*, 1949, **34**, 625) and Mosher *et al.* (*Anal. Chem.*, 1951, **23**, 1514) have been applied to the determination of Ga (0·02 to 0·5%) in HCl soln. obtained in the processing of brown coal. Bismuth was used as internal standard, with the line-pair Ga 2943 A – Bi 2938 A. It was found that results by the Schiebe and Rivas method had a mean coeff. of variation of ±10% as compared with ±4·8% for those by the rotating disc method. Full procedural details are given, and the purification of the graphite discs is described. R. E. E.

60. **Photometric determination of gallium and indium by means of quercetin.** I. P. Alimarin, A. P. Golovina and V. G. Torgov (M.V. Lomonosov Moscow State Univ.). *Zavod. Lab.*, 1960, **26** (6), 709–711.—*Procedure for Ga*—The soln. containing between 2·5 and 20 μg of Ga is treated with 1 ml of a 0·1% soln. of quercetin in ethanol, 5·5 ml of ethanol or methanol and 5 ml of an acetate buffer soln. (pH 4), and diluted to 25 ml. The extinction at 455 mμ is measured after 15 to 20 min. *Procedure for In*—The soln. containing between 10 and 100 μg of In is treated with 1 ml of 0·1% quercetin soln., 13·75 ml of ethanol and 5 ml of an acetate buffer soln. (pH 5), and diluted to 25 ml. The extinction at 416 mμ is measured after 10 to 15 min. Interference in the determination of Ga and In is caused by Al, F, oxalate, citrate and tartrate, but not by Zn or Cd at the relative concn. of Ga to Zn of 1:50, Ga to Cd ≃1:30, In to Zn ≃1:10, and In to Cd ≃1:10. G. S. Smith

61. **New gravimetric method for the determination of scandium.** T. I. Pirtea. *Rev. Chim., Bucharest*, 1960, **11** (6), 336–338.—The method is based on the reaction of Sc with mercaptobenzothiazole (I) (sodium salt) with the formation of a white insoluble cryst. ppt. of $Sc(C_7H_4NS_2)_2OH$. The reagent should be freshly prepared by dissolving 1 to 10 g of **I** in water, filtering off any residue, treating with N NaOH, adusting to pH 8 to 8·5 and diluting with water so that the content of **I** is 15 to 20%. *Procedure*—The soln. of scandium nitrate, sulphate or chloride (pH 5 to 6) (5 to 10 ml) is treated dropwise, at room temp. with 2 to 3 ml of reagent; pptn. is immediate, and the ppt. is filtered on a sintered glass crucible, washed with 1% aq. **I** soln., then 3 times with 2 to 3 ml of water, and dried for 30 min. at 110° to 115°. The determination can be carried out directly in the crucible by adding to it an excess of the reagent, followed by an aliquot of the sample soln. with stirring, filtering by gentle suction and washing and drying as described above. Micro determinations can be similarly carried out with a Emich filter-stick. Errors range from +0·5% on 80 mg to ±3·4% on 20 mg. H. Sher

62. **Accuracy of photometric determinations of ium as cerium(IV) - sulphato-complexes.** G. ttschalk and H. Bartsch (Osram G.m.b.H. Studgesellschaft, Berlin-Charlottenburg, Germany). *anal. Chem.*, 1960, **174** (6), 423–429.—The absorpn max. of the Ce - sulphato-complex (**I**) in N SO_4 is at 315 mμ. The suitability of the Hg lines 366 and at 436 mμ for photometry of **I** (14 μg 14 mg per 100 ml) is discussed. The H_2SO_4 ncn. must be between 0·8 and 1·5 N; the excess of sulphate should be ≯ tenfold. The extinction is stant at 20° ± 2° for >6 hr. Interference by I, Al, colourless alkali- and alkaline-earth-metal s, Sc, Y, Zn, Cd, Zr, Hf, Th and Sn is negligible. the presence of Sr, Ba and Pb, the pptd. sulphate st be removed. At 366 mμ, Co does not inter-e, Ni, La and Ti interfere slightly, and Fe^{III}, II, V^V and Mn strongly. At 436 mμ, Co, Ni, and Ti interfere. In the presence of PO_4^{3-}, ılts for Ce are low. Methods for eliminating erferences are given. J. P. Stern

63. **Radiochemical determination of cerium by iid - liquid extraction.** J. J. McCown and R. P. rsen (Chem. Engng Div., Argonne National Lab., mont, Ill., U.S.A.). *Anal. Chem.*, 1960, **32** (6), –599.—Cerium can be separated from all other portant fission products, without any precipitaas or determination of chemical yield, by the thod described. The Ce^{4+} are extracted with 2-ethylhexyl) hydrogen phosphate from a HNO_3 - rO_3 mixture, the organic layer is washed with same mixture and the Ce is then extracted from vith a HNO_3 - H_2O_2 mixture. Traces of Ru are n removed by fuming the sample to dryness h $HClO_4$ in the presence of added $RuCl_4$. The is then extracted with dilute HCl and the acid n. is either plated for β-counting or γ-counted a well-type scintillation counter. The coeff. of iation (10 analyses) on an irradiated uranium y was 1·4%. K. A. Proctor

64. **Spectrographic determination of rare-earth ments.** Sh. G. Melamed, S. M. Polyakov and G. Zemskova. *Zavod. Lab.*, 1960, **26** (5), 554–.—The sample (one part) is mixed with Y_2O_3 parts) and carbon powder (20 parts) or with CO_3 (19 parts) and carbon powder containing 25% of Sc (20 parts) and excited in a d.c. arc ween carbon electrodes. The total wt. of sample 0 mg. The spectrum obtained during complete poration of the sample (120 to 130 sec.) is asured. Line intensities of the rare-earth eleits are compared with the intensities of the Y or lines. G. S. Smith

65. **Continuous electrophoretic separations of coactive rare-earth mixtures. I. Separation of um-144, terbium-160 and thulium-170, and of um-144, europium-152 and ytterbium-169 in 5 M lactic acid.** Z. Pučar and Z. Jakovac (Inst. ljer Bošković, Zagreb, Yugoslavia). *J. Chromrraphy*, 1960, **3** (5), 477–481 (in English).—coradiographs of continuous electrophoretic arations and of discontinuous two-dimensional trochromatographic separations of the mixtures presented. Comparison with the activities of separated rare-earth elements shows that plete separation is achieved. In the electro-matographic separations, Ce, Tb and Eu are ngly adsorbed on the filter-paper. Advantages the continuous electrophoretic method are (*a*) the slower components moving in the direction the electric field are not contaminated by a reversibly adsorbed fraction of the faster components and absolute separation is achieved; (*b*) that carrier-free preparations are possible from very dilute solutions; and (*c*) that the method is particularly useful for hazardous radioactive materials since it is easily made automatic.

S. M. Marsh

66. **The determination of bound and free carbon dioxide. Micro-determination of mixed alkali carbonate and hydroxide.** E. Schulek and Á. Endröiné. *Magyar Kém. Foly.*, 1960, **66** (6), 211–213.—The interference of atmospheric CO_2 in the determination of weak acids and of dilute soln. of strong acids and alkalis is eliminated by the use as a sealing liquid of pentane containing propane and butane. It is established that the fading of the pink colour of phenolphthalein in even very dilute alkaline soln. is partly due to the rupture of lactone-type linkages in the phenolphthalein molecule, and it is recommended that a little more indicator should be added just before the end-point. The carbonate content of dilute alkali hydroxide soln. (0·1 to 0·01 N) can be accurately determined by means of $Ba(OH)_2$. R. E. E.

67. **Scintillation counting of carbon-14.** D. S. Jenkinson (Rothamsted Expt. Sta., Harpenden, Herts., England). *Nature*, 1960, **186**, 613–614.—The method is a development of previous work (Schram and Lombaert, *Anal. Chim. Acta*, 1957, **17**, 417; Greenfield, *Analyst*, 1958, **83**, 114; Steinberg, *Nature*, 1958, **182**, 740) and is based on the counting of pulses of light emitted by a plastic phosphor in contact with a soln. of the isotope. The method is suitable for handling large numbers of samples with sp. activity of 0·1 to 10 μC in vol. of 6 to 12 ml, and is not dependent on the chemical form of the carbon. Since it is usually necessary to add a dye to prevent loss of light from the test cell, the colour of the soln. being examined is not important.

G. S. Roberts

68. **Potentiometric determination of carbon in non-ferrous metals.** W. Fischer and H. Bastius (Forschungsinst. für Nichteisen-Metalle, Freiberg-Sachs, Germany). *Metall.*, 1960, **14** (5), 429–434.—The procedure of Fischer and Schmidt (*Anal. Abstr.*, 1957, **4**, 432) has been adapted to the determination of traces of carbon in copper, titanium, titanium alloys, vanadium, nickel and molybdenum. With titanium and titanium alloys a 5-fold excess of copper should be added as a flux.

T. R. Andrew

69. **Rapid analysis of silica in refractories and minerals. Improved method of sample preparation.** C. B. Belcher and L. B. Skelton (Broken Hill Proprietary Co. Ltd., Central Research Laboratories, Shortland, N.S.W., Australia). *Anal. Chim. Acta*, 1960, **22** (6), 567–570 (in English).—Previous methods are reviewed, and a simple method for the dissolution of the sample before subsequent colorimetric determination is proposed. The finely ground sample is sintered with Na_2O_2 in a platinum crucible at 500° ± 5° for 10 min. The mixture is extracted with water and then H_2SO_4. Excess of peroxide, which interferes, is removed with $KMnO_4$. The Si is determined photometrically at 813 mμ by the reduction of silicomolybdic acid to the blue complex with $Na_2S_2O_5$ and 1-amino-2-naphthol-4-sulphonic acid. A calibration curve is necessary. Beer's law is obeyed for samples (50 mg) containing 8 to 34% of Si. Interfering

PO_4^{3-} and Fe are masked with tartaric acid. Alumina, TiO_2 and Fe_2O_3 can be determined in the sample extract. R. M. S. HALL

70. **Rapid volumetric determination of silica in slags.** B. Bieber and Z. Večeřa (State Inst. of Materials and Technol., Brno, Czechoslovakia). *Hutn. Listy*, 1960, **15** (5), 397–398.—To 0·2 g of finely powdered sample in a platinum dish add HNO_3 (1:1) (20 ml), heat till nitrous fumes disappear, then add HNO_3 (1:1) (20 ml) and cool to 40° to 50°. Add NH_4F (5 g) and mix till the soln. is clear. Add KNO_3 (15 g) and filter-paper pulp and mix for 5 min. Filter off the pptd. K_2SiF_6 on a polyethylene filter and wash with satd. neutral KNO_3 soln. till free from acid. Transfer the ppt. to a flask containing 10 ml of neutral 40% $CaCl_2$ soln. and 200 to 250 ml of hot H_2O, mix, add Tashiro indicator (10 drops) and titrate with 0·2 N NaOH till the soln. is green. Deduct a blank. The procedure takes 15 to 20 min. and the error is $\gtrsim \pm 1\%$. J. ZÝKA

71. **Detection of lead by selective paper chromatography.** M. Ziegler (Anorg.-Chem. Inst., Univ. Göttingen, Germany). *Z. anal. Chem.*, 1960, **174** (5), 323–324 (in German).—Lead (20 μg) can be detected in the presence of Hg^{2+} (20 mg), Cu^{2+} (3 mg), Ag^+ (15 mg) and Bi^{3+} (0·6 mg) by transferring the sample soln. containing the metals as nitrates to a paper strip carrying a band of CdS and developing for 4 hr. with a solvent containing isopropyl alcohol (50 ml), H_2O (45 ml), acetic acid (1 ml) and ammonium acetate (5 g). Only Pb^{2+} pass through the CdS zone and are detected above it by spraying with $(NH_4)_2S$ soln. W. T. CARTER

72. **Use of EDTA for analysis of the active masses of an accumulator and of red lead.** N. A. Filippova and T. F. Dubrovskaya. *Zavod. Lab.*, 1960, **26** (6), 711–716.—*Determination of $PbSO_4$. Procedure* (*a*): *Na_2CO_3 method for all active masses*—The sample (0·5 g for discharged masses, and 2 to 2·5 g for charged active masses) is boiled with 100 ml of 10% Na_2CO_3 soln. for 30 min. The soln. is filtered, the insol. matter is washed with hot Na_2CO_3 soln., and then with hot water, and the filtrate, after acidification to methyl orange, is treated with 3 ml of HCl (1:1), then boiled, and treated with 10 to 15 ml of hot 10% $BaCl_2$ soln. After 1 to 1·5 hr. at 50° to 60° the ppt. is filtered off and washed with hot water until Cl^- are removed. The paper and ppt. are boiled with 40 ml of 0·1 N EDTA (disodium salt), 30 ml of 25% aq. NH_3 and 40 to 50 ml of water, with the addition of aq. NH_3 as necessary, until the ppt. is dissolved. The cooled soln. is treated with 15 ml of an ammoniacal buffer soln., 10 ml of Mg - EDTA soln. [prepared by mixing 50 ml of a 0·1 N soln. of a salt of Mg and 15 to 20 ml of a buffer soln. (nature and concn. not stated) and diluting to 250 ml] and some Eriochrome black T - NaCl (1:100) indicator, and then titrated with $MgSO_4$ soln. to the colour change from blue to reddish-violet. *Procedure* (*b*): *acid method for negative masses*—The sample (0·5 to 2·5 g) is treated with 15 to 20 ml of HCl (1:1) and 25 ml of water, and boiled with zinc filings. The transformation of $PbCl_2$ to Pb is complete when additional zinc filings remain bright for 2 to 3 min. The Pb is filtered off and washed. The filtrate is treated with 3 ml of HCl and the determination is completed as described above. *Determination of PbO_2 and total Pb in active masses*—The sample (0·2 g) is treated with 10 to 15 ml of 20% KI soln., 30 ml of 0·1 N EDTA (disodium salt) and 15 ml of 5% acetic acid soln. in a closed flask, and shaken until the PbO_2 is dissolved. The soln. is then titrated with 0·1 N $Na_2S_2O_3$ to give the content of PbO_2. The titrated soln. is mixed with 15 ml of 10% aq. NH_3, 15 ml of an ammoniacal buffer soln., 10 ml of Mg - EDTA soln. and some Eriochrome black T - NaCl (1:100) indicator, and titrated with 0·1 N $MgSO_4$ to give the content of Pb. G. S. SMITH

73. **Action of metavanadates on diphenylthiocarbazone [dithizone] and diphenylcarbazone. Application to determinations of lead in the presence of vanadates.** J.-M. Bloch and J. Lazare (Lab. de Toxicol., rue de la Prairie, Nancy, France). *Bull. Soc. Chim. France*, 1960, (6), 1148–1150.—Both sodium meta- and hypo-vanadate form with dithizone a coloured vanadium complex [$(dithizone)_2VO_3$], which is soluble in H_2O and ethyl methyl ketone and thus interferes with the semi-micro determination of Pb when the ratio of V to Pb in the sample soln. is >2. Moreover, V and Pb are both present in the $CHCl_3$ extract of dithizone, and it is not possible to apply a correction for the content of V. A complete and convenient removal of V from the sample soln. can be effected with diphenylcarbazone. *Procedure*—Adjust the sample soln. ($\simeq$80 ml, 20 to 200 μg of Pb) to pH 4, add 10% aq. Na citrate soln. (5 ml) and 10% aq. KCN soln. (2 ml), and again adjust the pH to exactly 3·8 with aq. 2·4% K H phthalate soln. (100 ml). Titrate the V with a 0·125% soln. of diphenylcarbazone in ethyl chloroacetate, withdrawing the organic layer after each addition (0·5 ml) of titrant until this increment no longer shows a rose colour. Adjust the pH of the V-free aq. layer to 9·5 (NH_4Cl - aq. NH_3 buffer) and determine the Pb by the usual dithizone procedure. The error for V is $\pm 5\%$. W. J. BAKER

74. **Photometric determination of titanium with tiron.** P. N. R. Nichols (Material Res. Lab., The Mullard Radio Valve Co. Ltd., Mitcham Junction, Surrey, England). *Analyst*, 1960, **85**, 452–453.—Certain disadvantages in the method of Yoe and Armstrong (*Anal. Chem.*, 1947, **19**, 100), in which tiron in a soln. buffered to pH 4·7 is used for the determination of Ti, appear to be due to the high pH value. The effect of lowering the pH was therefore examined, the extinctions after colour development being measured at 405 mμ. Results confirmed that a pH of 4·3 is necessary for maximum colour development, but also showed a "plateau" between pH 2·3 and 4·0. The decrease in sensitivity ($\simeq$15%) is offset by the advantages of working at a lower pH (3·0 $\pm$ 0·2). The use of a buffer is not necessary and the colour is stable for 24 hr. Beer's law is obeyed up to at least 80 μg of Ti per 100 ml, but the intensity of the colour is affected by change of temp. Any Fe^{3+} present up to 5 mg per 100 ml can be reduced with ascorbic acid or by warming with hydroxyammonium chloride soln. and pptn. of S which may accompany the use of sodium dithionite is thus avoided. A. O. JONES

75. **Spectrographic determination of niobium and tantalum impurities in titanium.** N. I. Tarasevich, K. A. Semenenko and N. F. Melëkhina. *Vestn. Moskov. Univ., Ser. Khim.*, 1960, II, No. 2, 64–68.—A systematic study was made of the conditions of volatilisation and entry into the discharge zone of an a.c. and a d.c. arc of small amounts of Nb_2O_5 and Ta_2O_5 in the presence of large amounts of TiO_2

a result of this investigation the following ethod is proposed for the simultaneous deter- nation of Nb (0·004 to 0·3%) and Ta (0·006 to 3%). Spectra are excited with a d.c. arc, at amp. and 220 V. The electrode gap is 2 mm. e standards and samples (≃5 mg), mixed with an ual wt. of carbon, are volatilised from the crater mm deep) of the lower graphite electrode; the per graphite electrode is cone-shaped (2 mm ane). Spectra are photographed. The line pairs ed are Nb 2950·88 - Ti 2958·28 and Ta 2714·67 - 2713·76 A. The calibration curves of log I_{Ta}/I_{Ti} and log I_{Nb}/I_{Ti} *vs.* log *C* are rectilinear after rrection for background. The method is tested th artificial mixtures of TiO_2 with Nb_2O_5 and $_2O_5$ and the results are tabulated. The coeff. variation is ≃8·2% for Nb and Ta concn. of 004% and 0·006%, respectively. K. R. C.

76. Determination of zirconium in titanium and alloys by the catechol violet reaction. Yu. A. ernikhov, B. M. Dobkina and E. I. Petrova. *wod. Lab.*, 1960, **26** (5), 529–531.—Improvements the method previously described (Chernikhov *et* , *Anal. Abstr.*, 1960, **7**, 440) are given. By an crease in the concn. of EDTA and a reduction in e amount of reagent, 5 μg of Zr can be determined the presence of 2·5 mg of Ti. With low concn. of (>1 μg per ml) the max. absorption occurs at 0 mμ (mol. extinction coeff. 25,000) instead of at 0 mμ. In alloys of titanium, Zr can be deter- ined directly after dissolution of the sample in l, and Mo, Al and Nb do not interfere. With ounts of Nb <100 μg a similar amount should added to the blank soln. G. S. Smith

77. Determination of hydrogen in oxygen-con- ning zirconium hydrides by the hydrogen-evolu- n method. M. E. Straumanis, C. S. Lin and . J. James (Dept. of Metallurg. and Chem. Engng, niv. of Missouri, Rolla, U.S.A.). *Z. anal. Chem.*, 60, **174** (5), 345–348 (in English).—The sample is ssolved in 3 N HF, the volume of the evolved drogen is measured, and the residue of ZrO_2 is llected, washed, dried and weighed. The hydro- n content is then calculated from the expression— = $[V - 491{\cdot}43(A - R)]/10{,}627$, where *Z* is the ight (g) of H in the sample, *V* is the vol. of H olved, *A* is the sample weight and *R* is the weight Zr in the sample as oxide. The hydrogen ntent of 100-mg samples containing 1 to 2 mg of can be determined with a precision of ≃±10%. e method is not applicable to samples into which drogen has diffused at low temperatures. W. T. Carter

78. Determination of oxygen in zirconium and zcaloys by emission spectrography in an argon nosphere. J. Artaud and C. Berthelot (Section Contr. Anal. des Matér., Centre d'Études cléaires, Saclay, S. et O., France). *Mém. Sci. Métall.*, 1960, **57** (5), 338–434.—The polished mple (0·1 to 0·2 g) is diluted with ≃ five times wt. of platinum and subjected to a d.c. arc charge between graphite electrodes in an atmo- ere of Ar at 400 torr; O is then liberated as CO. O line 7771·9 A is used for the determination, Ar line 7891·1 A being taken as the internal ndard. Standardisation is effected by arcing own amounts of ZnO. J. H. Waton

79. Liquid scintillation spectrometry for the alysis of zirconium-95 - niobium-95 mixtures and ncidence standardisation of these isotopes. J. D. Ludwick (General Electric Co., Hanford Atomic Products Operation, Richland, Wash., U.S.A.). *Anal. Chem.*, 1960, **32** (6), 607–610.—Mixtures of ^{95}Zr and ^{95}Nb at concn. ratios ranging from 50:1 to 2:1 can be analysed by liquid scintillation spectrometry. The results obtained agree well with those by other methods, and no correction for self-absorption is required. K. A. Proctor

80. Separations involving sulphides. XII. Separation of thorium or titanium from some elements that form thio-salts. I. K. Taimni and S. N. Tandon (Chem. Lab., Univ. of Allahabad, India). *Anal. Chim. Acta*, 1960, **22** (5), 437–438.—Sodium sulphide and HCl are added to the test soln., the pptd. sulphide is filtered off and weighed, and the Ti or Th in the filtrate is determined as the oxide or 8-hydroxyquinolinate, respectively. By this procedure As, Sb, Te, Se, Mo, Hg, Au, Pt and Re can be separated from Th, with an accuracy within 0·75%, and Sb, Te, Se and Hg from Ti to within 0·3%. R. M. S. Hall

81. Spectroscopic determination of nitrogen-15 by means of a step filter. H. Faust (Agric. Chem. Inst., "Friedrich Schiller" Univ., Jena). *Z. anal. Chem.*, 1960, **175** (1), 9–18 (in German).—A detailed procedure is given. The precision of the method for samples containing 0·36 to 10% of ^{15}N is within ±5%. Results are compared with those of the mass-spectrometric method. B. B. Bauminger

82. Analysis of nitrogen for small concentrations of carbon monoxide by mass spectrometry. G. P. Schacher, R. E. Rippere and J. A. Hill (Gen. Engng Lab., General Electric Co., Schenectady, New York, U.S.A.). *Appl. Spectroscopy*, 1960, **14** (3), 79–80.—Because of the coincidence of the major peaks of N_2 and CO at mass 28, the determination of CO in combustion gases becomes difficult when the content is below 5%. By the use of Schütze reagent (prepared from silica gel, I_2O_5 and H_2SO_4), the CO may be converted into CO_2 and can thus be readily detected at concn. of 0·1%. A more accurate determination of the CO_2 obtained by the conversion from CO may be made by freezing out water and CO_2 from the sample before passage through the reagent. P. T. Beale

83. Determination of nitrogen in boron nitride. J. D. Cosgrove and E. C. Shears (Borax Consolidated Ltd., Cox Lane, Chessington, Surrey, England). *Analyst*, 1960, **85**, 448–449.—The sample (0·2 g) is fused with LiOH in a silver thimble enclosed in a test-tube carrying an inlet tube for the carrier gas (N) and an outlet tube leading the evolved NH_3 into 2% H_3BO_3 soln. The test-tube is immersed in sand and is heated to 500° to 550°. After 30 min. the H_3BO_3 soln. is titrated to methyl orange indicator with 0·25 N HCl, and the titration is continued at 10-min. intervals until there is no increase of titre during 30 min. Alternatively, the sample is heated with 80% H_2SO_4 in a sealed Carius tube at 320° for 8 hr. The contents of the tube are washed into a distillation flask, 45% NaOH soln. is added to the diluted liquid and the evolved NH_3 is distilled into 4% H_3BO_3 soln., which is finally titrated with 0·25 N HCl. The titre is corrected by means of a blank determination. Hydrolysis by 80% H_2SO_4 is the better method, but fusion with LiOH has the advantage that N and B may be determined in the same 0·2-g sample. A. O. Jones

84. **Application of pyramidon [amidopyrine] to detection of nitrous acid.** W. Wawrzyczek (General Chem. Dept., School of Agric., Olsztyn, Poland). *Nature*, 1960, **186**, 883.—When a few drops of a 5% soln. of amidopyrine and a few drops of dil. acetic acid are added to soln. containing NO_2^- in concn. as low as 0·009 mg per ml, a violet colour is formed which is not extracted by ether, $CHCl_3$, amyl alcohol or CCl_4. The reaction, which may be accelerated by heating, is not affected by sulphuric, nitric, iodic, hydrochloric, phosphoric, boric, oxalic, citric, tartaric or hydrobromic acid or by some bi- and ter-valent metal ions. K. A. Proctor

85. **Spectrophotometric determination of phosphate using lanthanum chloranilate.** Kiyoko Hayashi, Taeko Danzuka and Keihei Ueno (Dojindo & Co. Ltd., Kumamoto, Japan). *Talanta*, 1960, **4** (4), 244–249 (in English).—Phosphate reacts with lanthanum chloranilate to form insoluble lanthanum phosphate and free chloranilic acid which is determined by direct spectrophotometry on the filtered soln. *Procedure*—To the sample soln., containing 0·15 to 5 mg of phosphate, add 0·03 M Na succinate (5 ml), 95% ethanol (25 ml) and lanthanum chloranilate (0·1 g). Dilute the mixture to 50 ml and shake mechanically for 10 min. Filter a portion of the soln. through paper and measure the extinction at 530 mμ against a reagent blank. Read off the phosphate concn. from a calibration curve. Almost all cations interfere and must be removed by ion exchange; NO_3^- and Cl^- in concn. up to 400 p.p.m. do not interfere; SO_4^{2-} interfere but the effect can be eliminated by adding 800 p.p.m. of SO_4^{3-} to the sample and standards; F^- interfere seriously. The procedure has a precision of $\simeq \pm 2\%$. W. T. Carter

86. **Isotope dilution procedure for the determination of the true phosphate content of rock phosphates.** J. Rijkheer (Research Dept., African Explosives & Chem. Ind., Ltd., P.O. Northrand, Transvaal). *J. S. Afr. Chem. Inst.*, 1960, **13** (1), 1–6.—In the procedure described, accurately weighed samples of standard $Mg_2P_2O_7$ labelled with ^{32}P (prep. described) were digested with the rock samples in acid soln., the soln. was passed through a column of Amberlite IR-120 resin (H^+ form) and phosphate in the percolate was isolated as NH_4MgPO_4, and ignited to $Mg_2P_2O_7$. The specific activity of this material was then compared with the standard, and the phosphate content of the rock was calculated in the usual way. All the phosphates must be in the ortho form. To ensure high accuracy the experimental procedure described must be closely followed. A. M. Spratt

87. **Quantitative analysis of alkali polyphosphates.** O. Pfrengle (Forsch.-lab. der Chem. Fabrik Budenheim Rudolf A. Oetker KG, Wiesbaden). *Fette, Seif., Anstrichmitt.*, 1960, **62** (5), 433–439.—Various methods of determining the mean chain-length of a polyphosphate glass are compared. These include the Samuelson titration value (*Svensk. Kem. Tidskr.*, 1944, **56**, 343), the Na_2O to P_2O_5 ratio, and a paper-chromatographic method. Diagrams of typical chromatograms are given. (28 references.) H. M.

88. **Amperometric determination of arsenic and antimony.** V. A. Zakharov, O. A. Songina and N. A. Dragavtseva (Kazakh State Univ.). *Zavod. Lab.*, 1960, **26** (5), 537–540.—Two aliquots (5 or 10 ml) of the sample soln. are neutralised with NaOH soln. (or $NaHCO_3$) and treated with 0·5 to 1 g of $NaHCO_3$, followed by dilution to 25 or 30 ml. In one soln. the sum of As and Sb is determined by titration with 0·01 N iodine at +0·2 V *vs.* the S.C.E. with a rotating platinum electrode. The other soln. is treated with an amount of 0·01 N K_2CrO_4 equivalent to 1·5 times the amount of iodine soln. needed in the first titration, and the As is titrated with 0·01 N iodine after ≥ 30 sec. G. S. Smith

89. **Complexometric determination of arsenates.** R. Vasiliev and G. Anastasescu. *Rev. Chim., Bucharest*, 1960, **11** (5), 298.—The proposed method is based on an indirect determination by means of Bi^{3+} in acid medium. *Procedure*—A soln. of the sample (0·3 g) in a few ml of water (containing 0·5 to 1 ml of 10% HNO_3 for water-insoluble samples) is treated with 40 ml of 0·05 M $Bi(NO_3)_3$ and diluted to 100 ml. The mixture is filtered; the first 20 to 30 ml is rejected, and the next 50 ml is treated in a titration flask with 0·5 ml of a 0·1% soln. of catechol violet as indicator and titrated with 0·05 M EDTA (disodium salt) until the first colour-change to violet. After the addition of 200 ml of water, titration is continued until the colour changes to yellow; NH_4^+, Li^+, Mg^{2+}, Ca^{2+}, Sr^{2+}, Ba^{2+}, Al^{3+}, Zn^{2+}, Mn^{2+}, Co^{2+}, Ni^{2+}, Cd^{2+}, Cu^{2+}, Pb^{2+}, UO_2^{2+} and Ag^+ do not interfere; Fe^{3+}, Ga^{3+}, In^{3+}, Zr^{4+}, Th^{4+} and Hg^{2+} do. Perchlorates do not interfere, but Cl^-, SO_4^{2-}, PO_4^{3-} and $Cr_2O_7^{2-}$ do. A satisfactory method of eliminating Cl is the addition of 10 ml of a 10% soln. of $AgNO_3$ before the addition of the $Bi(NO_3)_3$ soln. Similarly 10 ml of a 10% soln. of $Ba(NO_3)_2$ eliminates SO_4^{2-}. Errors do not exceed those normally found in complexometric methods. H. Sher

90. **Determination of antimony and tin in type metals by means of X-ray spectrography.** A. J. Rossouw. *S. Afr. Ind. Chem.*, 1960, **14** (3), 44–46.—The X-ray spectrograph has been used to determine the Sb (11 to 16%) and Sn (4 to 10%) contents of antimony - tin - lead alloys. Samples were chill-cast from the melt to fit the spectrograph holder; pure lead was used to obtain a background value, and a standard alloy was used to obtain a calibration curve. A determination may be completed in 5 min. and results are estimated to be accurate to ±0·2%. T. R. Andrew

91. **New method for the colorimetric determination of bismuth.** E. Popper, V. Junie, L. Popa and L. Roman (Fac. of Pharm., Cluj). *Rev. Chim., Bucharest*, 1960, **11** (6), 341–343.—The proposed method is based on the pptn. of Bi^{3+} with 5-anilino-2-mercapto-1,3,4-thiadiazole (**I**), dissolution of the ppt. in an excess of **I**, and intensification of the colour by the addition of KI. Standards are prepared from a soln. containing 0·1256 g of Bi in 100 ml of 5 N HCl; aliquots of 0·005 to 2 ml are treated with 6 ml of fresh 1% ethanolic **I** soln. and 2 ml of 25% aq. KI soln., and diluted to 10 ml with 96% ethanol. The colours are read after 1 min. in a Lange photometer model N1 and the results are plotted. For larger quantities a soln. containing 2·5117 g of Bi per 100 ml is used, since pptn. occurs if more than 2 ml of test soln. is taken. Two curves are prepared covering the ranges 6 to 480 μg and 0·95 to 50 mg, respectively. Unknown samples are treated in the same way. Errors range from +3% to −0·2%. The sensitivity is 6 μg per ml. It is claimed that the method can be adapted for the determination of Bi in the presence of other heavy metals; NO_3^- and oxidising ions interfere. H. Sher

2. **Amperometric complexometric determination bismuth in easily fusible alloys [containing cadm, tin and lead].** Yu. I. Usatenko and M. A. kina (F. É. Dzerzhinskiĭ Chem.-Tech. Inst., iepropetrovsk). *Zavod. Lab.*, 1960, **26** (5), 542–.—The sample (0·5 to 0·6 g) is dissolved by boilwith 5 to 6 ml of HNO_3 (1 : 1), the soln. is treated h 4 to 5 ml of 0·5 M H_2SO_4 and evaporated to ipient fuming; 6 to 7 ml of HNO_3 (1 : 1) is added, soln. is boiled for 1 to 2 min., three times its . of water is added, the soln. is again boiled, and cooled soln. is diluted to 250 ml. An aliquot ml) is treated with 3 g of KNO_3 and the soln. titrated with 0·05 M EDTA (disodium salt) at ·9 V *vs.* the S.C.E. with a platinum electrode.

G. S. Smith

3. **Colorimetric determination of bismuth and d in copper and alloys.** R. Socolovschi. *Rev. m., Bucharest*, 1960, **11** (6), 348–349.—The sample lissolved in HNO_3, metals that give ppt. with aq. I_3 and KCN are complexed with citrate, and Bi l Pb are extracted together with dithizone. They then re-extracted into dil. HNO_3 and determined cessively with dithizone; Tl^+, which interfere, usually absent, and Sn^{2+}, which also interfere, oxidised by the HNO_3. *Procedure*: (*i*) *extraction Bi and Pb*—The sample (1 g) is dissolved in the nimum of conc. HNO_3 and filtered. Oxides of N boiled off, the soln. is slightly diluted with ter, treated with 40% ammonium citrate soln. ml) and neutralised with conc. aq. NH_3; 20% CN soln. is added slowly to the cooled soln. till mplexing is complete, a further 1 ml of KCN soln. added and the soln. is brought to pH 7·5 to 8 enol red). The mixture is then extracted with 03% dithizone soln. in $CHCl_3$ till the extract colourless, and the Bi and Pb are re-extracted o 10 ml of N HNO_3. (*ii*) *Determination of Bi*—e HNO_3 extract is adjusted to pH 2 with aq. I_3 and extracted with 0·001% dithizone soln. in CCl_3 till the extract remains green. The comed extracts are then treated in a stoppered tube h more dithizone soln. in small portions till the our becomes grey. A second tube containing same amount of dithizone soln. is then titrated h standard Bi soln. (5 μg per ml in 0·01 N HNO_3) the same grey colour. The error for $\simeq$0·001% Bi is $\pm$10%. (*iii*) *Determination of Pb*—The NO_3 extract after removal of Bi is neutralised to nus paper with aq. NH_3 and treated with alkaline N - citrate soln. (50 ml of 5% KCN soln., 15 ml 5% ammonium citrate soln., 5 ml of conc. aq. I_3 and 450 ml of water) (20 ml). Dithizone soln. $CHCl_3$ (0·003%) is added from a burette till a let colour is obtained, and the excess of dithizone removed by washing with alkaline KCN soln. ml of 5% KCN soln., 5 ml of conc. aq. NH_3 and ml of water) (2 × 30 ml). The org. layer is n suitably diluted with $CHCl_3$ and the extinction ead in a Pulfrich photometer with a green filter. e error for $\simeq$0·005% of Pb is $\pm$20%.

R. E. E.

4. **Complexometric titrations (chelatometry). VII. Micro-determination of bismuth and lead stepwise titration, with xylenol orange as icator. Study of indicator colour changes with the of photometric titration.** S. Kotrlý and J. štál (Inst. Chem. Technol., Pardubice-Rybitví, choslovakia). *Coll. Czech. Chem. Commun.*, 0, **25** (4), 1148–1164 (in German).—*Bismuth*—a solution containing 0·004 to 0·08 mg of Bi photometric titration) or >0·1 mg of Bi (for visual titration) in 30 ml of $\simeq$0·1 N HNO_3 add xylenol orange soln. and titrate with 0·001 M EDTA (disodium salt) (**I**) photometrically at 530 mμ (measuring the decrease of extinction) or visually till the red colour changes to yellow. Chlorides and bromides interfere. *Lead*—To a soln. containing 0·004 to 0·08 mg of Pb (photometric titration) or >0·1 mg of Pb (visual titration) in 30 ml of the soln. at pH 5 (addition of hexamine) add xylenol orange and titrate with 0·001 M **I** photometrically at 550 mμ or visually to a red - violet colour.

J. Zýka

95. **Analysis of bismuth - antimony - tellurium - selenium combinations.** J. F. Reed (Technol. Dept., Westinghouse Res. Lab., Pittsburgh, Pa., U.S.A.). *Anal. Chem.*, 1960, **32** (6), 662–664.—Procedures are fully described for the successive determination of the elements named in a single sample. Selenium is pptd. as the free element with satd. aq. SO_2 soln. from a soln. of the sample in conc. HCl; the filtrate is diluted to 3 M in HCl. Tartaric acid is added to retain Sb in solution, and elementary Te is pptd. with SO_2 soln. and hydrazine hydrochloride soln. The filtrate is divided into two parts. In the first, Sb and Bi are pptd. with H_2S, the ppt. is fumed with H_2SO_4 and HNO_3, diluted with water, and reduced with SO_2 soln. The excess of SO_2 is boiled off, HCl is added, and the Sb is titrated potentiometrically with 0·1 or 0·05 N $KMnO_4$. The second portion is evaporated to dryness, the residue is re-dissolved in the minimum of N HNO_3, and diluted to 50 ml, and the Bi is titrated with 0·01 M EDTA (disodium salt) at pH 2·4 $\pm$0·1, with thiourea as indicator. The accuracy and precision are within 2 parts per thousand.

R. E. E.

96. **Precise method for determining vanadium in essentially pure vanadium metal.** W. C. Dietrich (Union Carbide Nuclear Co., Y-12 Plant, Oak Ridge, Tenn.). *U.S. Atomic Energy Comm.*, Rep. Y-1294, Mar., 1960. 10 pp.—Precision resulting in a limit of error (P = 0·95) for a single determination of $\pm$0·079% is achieved by oxidising the weighed and dissolved sample to V^V and allowing this soln. to react with a weighed slight excess of $(NH_4)_2SO_4.FeSO_4.6H_2O$. The excess of reagent is then back-titrated with standard $K_2Cr_2O_7$ soln.

Nucl. Sci. Abstr.

97. **A new reducing agent. Reduction of vanadium(IV) and uranium(VI) with ferrous ion in catechol solutions.** J. W. Miller (Res. Div., Phillips Petroleum Co., Bartlesville, Okla., U.S.A.). *Talanta*, 1960, **4** (4), 292–293 (in English).—Ammoniacal soln. of V^{IV} and U^{VI} containing catechol are rapidly reduced by Fe^{2+} to V^{III} and U^{IV}, respectively. Potentiometric titrations may be carried out with a soln. containing M NH_4Cl, M aq. NH_3, 0·1 M Na_2SO_3 and 4% of catechol, with a mercury pool *vs.* a silver - silver chloride electrode; V^{IV} and U^{VI} cannot be differentiated and the V^{IV} must be removed before titrating the U^{VI}.

W. T. Carter

98. **Determination of tantalum and tungsten in rocks and meteorites by neutron activation analysis.** D. H. F. Atkins and A. A. Smales (A.E.R.E., Harwell, Berks., England). *Anal. Chim. Acta*, 1960, **22** (5), 462–478.—After irradiation and dissolution of the sample and a standard in the presence of carriers, W and Ta are co-pptd. by tannin and cinchonine, and the ppt. is converted into the oxides and dissolved in HF - H_2SO_4. Tantalum is separated by solvent extraction with isopropyl ketone

or isobutyl methyl ketone, and finally converted into the tri-2,2′-dipyridyl ferrous fluorotantalate for counting; W is recovered from the aq. phase, and counted as the 8-hydroxyquinolinate. The activity is measured by either β- or γ-counting. The radiochemical purity of the final source may be checked by half-life measurements, β-absorption curves and γ-spectra. Errors caused by alternative nuclear processes on W, Re and Os appear to be negligible. When standard samples were analysed the results were within $\pm 10\%$ for Ta levels of 0·02% and within 0·05 to 0·2% for W. Results of duplicate analyses agreed to within $\pm 10\%$. The limits of sensitivity attainable are 10^{-10} g for Ta and 10^{-11} g for W. Measurements have been made down to 8×10^{-10} g of Ta and 2×10^{-9} g of W with ^{182}Ta and ^{187}W.

R. M. S. Hall

99. **Determination of ultra-micro amounts of sulphate as methylene blue. I. The colour reaction.** L. Gustafsson (Dept. of Anal. Chem., Univ., Uppsala, Sweden). *Talanta*, 1960, **4** (4), 227–235 (in English).—Optimum conditions are outlined for the colorimetric determination of H_2S by conversion into methylene blue with *p*-aminodimethylaniline and Fe^{3+}. *Procedure*—Add the sample soln. (up to 70 ml) to a soln. 0·25 M in Zn acetate and 0·1 M in Na acetate (10 ml) and place in a thermostat at 20° for 10 min. Add *p*-aminodimethylaniline sulphate soln. (0·005 M in 3·5 M H_2SO_4) (10 ml), mix and add $(NH_4)_2SO_4.FeSO_4$ soln. (0·25 M in 0·5 M H_2SO_4) (2 ml). Dilute to 100 ml and measure the extinction at 667 mμ and 20° after 15 min. The reaction is sensitive to the acid concn. (optimum 0·3 M), but the concn. of the other reagents are not critical. Both the extinction and the yield are temperature-dependent, but once the colour is formed it is stable for several hours. Traces of Cu and Hg interfere seriously; de-ionised water must be used for all reagents and the Zn acetate soln. must be freed from heavy metals by pptn. with Na_2S. Beer's law is obeyed up to 25 μg of S, and the yield of methylene blue is $\simeq 65\%$ of the theoretical.

II. The reduction. L. Gustafsson. *Ibid.*, 1960, **4** (4), 236–243.—The sulphate is reduced to H_2S with NaH_2PO_2 and HI in a stream of N, and the H_2S is collected in Zn acetate soln. and determined colorimetrically as methylene blue, as described in Part I. *Procedure*—Place the solid sample containing up to 100 μg of S in a reduction apparatus (illustrated) consisting of a flask with a side arm, a reflux condenser and a delivery tube fitted with a gas washer containing water. Place the delivery tube in a flask containing water (68 ml) and Zn acetate - Na acetate soln. (10 ml). Add the reducing soln. [$NaH_2PO_2.H_2O$ (2·5 g), glacial acetic acid (25 ml) and HI (100 ml)] (3 ml) and pass N through the apparatus. Heat the contents of the flask rapidly to boiling and reflux for 10 min. Disconnect the flask and complete the determination as described in Part I. The presence of water in the sample causes low results and aq. samples must be evaporated to dryness before analysis. Nitrate ions and other oxidising agents interfere, but Fe^{III} (up to 20 mg), alkali metals and alkaline-earth metals do not. The standard deviation for a single result for 2 μg of S was $\pm 0{\cdot}05$ μg (9 determinations).

W. T. Carter

100. **Determination of sulphate in chromium plating solutions.** G. J. McKinley and A. Q. Jones (Anal. Control Group, Van der Horst Corp., Olean, N.Y.). *Plating*, 1960, **47** (6), 634–636.—By reduction of SO_4^{2-} to S^{2-} and titration with KIO_3 - KI reagent, $\not<2$ g of SO_4^{2-} per litre is determined with an accuracy within $\pm 1\%$; Fe and Cr up to 8 g per litre do not interfere. To prepare the reducing mixture, heat 200 ml of HI (sp. gr. 1·5 to 1·7) and 200 ml of HCl (sp. gr. 1·18) with 50 ml of H_3PO_2 soln. (50%) under reflux for 1 hr. under N. Store at $\not>0°$. *Procedure*—Add 5 ml of test soln. to 30 ml of reducing mixture in a 100-ml flask, passing N until all iodine fumes are removed. Connect the flask to a vertical condenser, and the top of this to a delivery tube dipping into a flask which contains 150 ml of H_2O and 10 ml of $ZnSO_4$ soln. (50 g of $ZnSO_4.7H_2O$ in 250 ml of H_2O and 250 ml of conc. aq. NH_3; allow to settle and filter). Adjust the flow of N to 2 or 3 bubbles per sec. and heat the contents of the flask under reflux for 1 hr. Add starch soln. to the receiving flask and titrate with KIO_3 - KI soln. (0·7134 g of KIO_3 and 8 g of KI in H_2O to make 1 litre) to a permanent blue end-point.

P. D. Parr-Richard

101. **[Spectrographic] determination of silicon and other impurities in selenium.** V. L. Ginzburg and N. P. Glukhovetskaya (N.S. Kurnakov Inst. of Gen. and Inorg. Chem., Acad. Sci., USSR). *Zavod. Lab.*, 1960, **26** (5), 559–561.—Standards for silicon are obtained from a selenium alloy containing 3% of Si, which is first fused *in vacuo* at 750° to 800° for 3 hr., then mixed with pure Se to reduce the content of Si to 0·3% and fused *in vacuo* at 400°. Further dilution is carried out similarly to give a range of standards. Samples and standards in cavities in carbon rods are excited in an a.c. arc. In the spectrum the line Se 2549·19 A is compared with one of the following lines of Si—2516·12 A for contents between 10^{-4} and 3×10^{-3} %, 2881·58 A for 2×10^{-4} to 2×10^{-2} %, 2514·33 A for 10^{-3} to 5×10^{-2} %, or 2435·16 A for 10^{-2} to 3×10^{-1} %. Determinations of Cu, Pb, Mg, Al, Ag, Fe, As, Sb, Ni, Bi, Te, Cd, Hg, Sn and Ni are carried out by means of standards, obtained by dilution with Se of an alloy of known composition containing larger amounts of these impurities.

G. S. Smith

102. **Separation of tellurium and iodine in different oxidation states by oxidation - reduction resins.** R. Münze (Zentralinst. Kernphysik, Dresden, Germany). *J. prakt. Chem.*, 1959, **7**, 262–267.—When a TeO_3^{2-} soln. was added to a cation-exchange resin (Sn^{2+} form) the Te was retained on the column as the element. The reduction capacity was 9 mg of Te^{IV} per g of resin. The Te was not eluted with H^+ in the pH range 1·1 to 6·1, but was with HCl of concn. $>$N. The behaviour of aq. soln. of iodine, I^- and IO_3^- on the Te - Sn^{2+} column showed that the iodine species could be eluted free from Te. The continuous separation of ^{132}I species from the β-emitting ^{132}Te reduced on the Sn^{2+} column should be possible by this method. The IO_3^- were not reduced.

Chem. Abstr.

103. **Determination of traces of lead in tellurium acid.** C. R. Veale and R. G. Wood (D.S.I.R. National Chem. Lab., Teddington, Middx., England). *Analyst*, 1960, **85**, 371–374.—For the preparation of the standard graph, 0 to 2·5-ml portions of standard lead soln. (4·17 μg of Pb per ml) are extracted by a modification of the procedure described by Sandell ("Colorimetric Determination of Traces of Metals," 3rd Ed., Interscience Publishers Inc., New York, 1959, p. 555) for isolating Pb. The soln. in HNO_3 is treated with an a

I_3 - cyanide - sulphite soln. (*loc. cit.*) and 25 ml of oln. of dithizone in CCl_4. The extinction of the ered CCl_4 layer is measured at 520 mμ. A table wt. of the sample (up to 15 g) is dissolved water, and HCl is added to adjust the pH to to 2·0 according to the wt. of sample. The uid is heated to b.p. after addition of hydrazine drate soln. and is set aside for ⩽3 hr. The pH ould then be 1·6 to 2·1. The supernatant liquid removed through an immersion filter, the ppt. is shed, dissolved in HNO_3 and treated as in eparation of the standard graph. The extinction the final soln. is measured at 520 mμ against a gent blank prepared by treating 5 ml of conc. NO_3 similarly. A. O. Jones

104. **Oxidations with potassium permanganate the presence of fluoride. III. Determination of romium(III) and vanadium(IV).** I. M. Issa and Hamdy (Cairo and Assiut Univ., Egypt). *Z. al. Chem.*, 1960, **174** (6), 418–422 (in English).— e Cr^{III} or V^{IV} is oxidised within 5 min. by excess $KMnO_4$ in NaOH soln. (0·3 to 1·8 M). Acidifica- n with H_2SO_4, addition of 2% NaF soln. and then excess of standard Hg^I soln. and back-titration the excess with $KMnO_4$ at 40° in the presence of $^{2+}$ permit the determination of the $KMnO_4$ sumed without interference by Cr^{VI} and V^V; to 4·5 mg of Cr^{III} and 50 mg of V^{IV} can be deter- ned with an accuracy within ±0·5%. J. P. Stern

105. **Determination of micro amounts of phos- orus in metallic chromium.** A. A. Fedorov and V. Linkova. *Zavod. Lab.*, 1960, **26** (5), 535–536. The sample (1 g for contents between 0·002 and 1% and 2 × 1·25 g for contents between 0·0002 d 0·002%) is placed in a 1-litre beaker (or in two itre beakers) and dissolved in 70 ml of HCl (1:1) d. with Br, an excess of Br being maintained roughout the dissolution. The soln. is evaporated a syrup and then treated with a purified 3% soln. KOH (500 ml in excess after neutralisation of the d), prepared by treating 1 litre with 20 ml of , $Ca(NO_3)_2.4H_2O$ soln., setting aside for 2 hr., ling a further 8 ml of 4% $Ca(NO_3)_2.4H_2O$ soln., d filtering after 20 min. The soln. is treated with ml of 30% H_2O_2 soln. [purified by shaking with shly pptd. $Mg(OH)_2$], boiled for 20 min., treated the cold with 8 ml of 4% $Ca(NO_3)_2.4H_2O$ soln., l set aside for 2 hr. After addition of a further l of the $Ca(NO_3)_2$ soln. and setting aside for 20 n. the ppt. is collected and washed with the KOH n. It is then dissolved in 50 ml of hot HNO_3 9), the filter being washed with 30 to 40 ml of HNO_3 (1:3) and then with hot water. The n. is evaporated to dryness and the residue is solved in 10 ml (or 2 × 5 ml) of conc. HNO_3 l 15 to 20 ml of water. After addition of 2 ml 4% $KMnO_4$ soln. [purified by boiling 300 ml for in. with 4 g of $Fe(NO_3)_3.9H_2O$ and filtering after min.], the soln. is boiled to precipitate MnO_2, ich is then dissolved with a few drops of 5% NO_2 soln. Oxides of N are boiled off and the $_4{}^{3-}$ in the cooled and diluted soln. are deter- ned colorimetrically by the molybdenum blue ction. G. S. Smith

106. **Complexometric determination of molyb- num.** J. B. Headridge (Dept. of Chemistry, The iv., Sheffield, England). *Analyst*, 1960, **85**, –381.—To the approx. neutral soln. of molyb- e (≡ 1 to 11 mg of Mo), adjusted to ≃10 ml, 1 ml each of conc. HCl and a saturated soln. of hydrazine sulphate are added and the mixture is maintained at 95° for 5 min. To the cooled liquid is added 10 ml of EDTA soln. (≃0·93 g of the di- sodium salt per 250 ml) and the mixture is trans- ferred to a sample holder for an E.E.L. titrator or other filter photometer or spectrophotometer adapted for photometric titration. A buffer soln. (5 ml of a 45·9% soln. of Na acetate trihydrate) and alizarin complexone indicator (1,2-dihydroxy- anthraquinon-3-ylmethylamine-*NN*-diacetic acid) are added, and with the use of an Ilford No. 604 filter the galvanometer reading is set to ≃8 and the soln. is titrated with standard $ZnCl_2$ soln. The end-point is ascertained from the titration graph and corrected for a blank determination. The procedure described will require modification if metals other than alkali or alkaline-earth metals are present. A. O. Jones

107. **Cerimetric determination of molybdenum in high-chloride media using the molybdenum blue reaction.** C. M. Callahan, S. C. Foti and J. R. Lai (U.S. Naval Radiological Defense Lab., San Fran- cisco, Calif.). *Anal. Chem.*, 1960, **32** (6), 635–637.— The neutral test soln. is acidified with an equal vol. of 2 N HCl and passed through a Jones reductor (previously washed with N HCl) at a rate of 20 to 30 drops per min. The percolate and washings are received under the surface of the receiver soln., or allowed to drop through an atmosphere of N. The receiver soln. should contain at least a 16-fold excess of Na_2MoO_4, and be at a pH such that an equiv. amount of molybdenum blue is formed; 5·5 to 6 g of $Na_2MoO_4.2H_2O$ in 100 ml of N HCl is satisfactory for up to 4·5 milli-equiv. of Mo. The soln. is then slowly titrated with standard $Ce(SO_4)_2$ soln. to a colourless end-point. The method is slightly less precise than the permanganate method, and the titration time is longer, but the $Ce(SO_4)_2$ soln. is very stable, it can be used in media con- taining much chloride, when the permanganate method is inapplicable, and no indicator correction is required. R. E. E.

108. **Chromometric determination of sexavalent molybdenum in the presence of thiocyanates.** A. I. Busev and Gȳn Li. *Vestn. Moskov. Univ., Ser. Khim.*, 1960, II, No. 2, 73–75.—The potentiometric titration of Mo^{6+} in HCl soln. with $CrCl_2$ in the presence of KSCN is studied with the use of a platinum indicator electrode. Mixtures of $(NH_4)_2MoO_4$ soln. (≃0·00959 g of Mo) (10 ml) and 6·1 N HCl (70 ml) are treated with CO_2 for 25 min.; KSCN (1 g) is then added and CO_2 is passed for another 5 min. The results for the titration at room temp. are tabulated. K. R. C.

109. **Photometric determination of molybdenum in the presence of tungsten by means of sodium 5-phenyl-2-pyrazoline-1-dithioformate.** A. I. Busev, V. M. Bȳr'ko and I. I. Grandberg. *Vestn. Moskov. Univ., Ser. Khim.*, 1960, II, No. 2, 76–80.— The complex of Mo with Na 5-phenyl-2-pyrazoline- 1-dithioformate (**I**) is very stable and Beer's law is obeyed in the concn. range 0 to 8 μg per ml. With max. absorption at 530 mμ, the mol. extinction coeff. of the Mo - **I** complex extracted with $CHCl_3$ is 3200 and the detectable minimum is 0·6 μg per ml. At 530 mμ, Fe^{3+}, Cu^{2+}, Mn^{2+} and V interfere; W does not interfere as it does not form a coloured complex with **I**. K. R. C.

110. **Analytical chemistry of benzoin α-oxime complexes of molybdenum, tungsten and vanadium.** H. J. Hoenes and K. G. Stone (Michigan State Univ., East Lansing, U.S.A.). *Talanta*, 1960, **4** (4), 250–263 (in English).—Sexavalent molybdenum is pptd. by benzoin α-oxime as $MoO_2(C_{14}H_{12}O_2N)_2$ from acid soln. in the range from pH 2 to 5% of H_2SO_4 by volume. Interference from Cr^{III} and V^{IV} is prevented by reduction with $(NH_4)_2SO_4.FeSO_4$ and that from Fe^{II} and Fe^{III} by washing the ppt. with 1% H_2SO_4; W^{VI} interferes and must be absent. The complex can be weighed after drying at 105°. In the solvent extraction of small amounts of the complex into $CHCl_3$, interference from V^V and W^{VI} is prevented by the addition of KH_2PO_4 and $(NH_4)_2SO_4.FeSO_4$. Sexavalent tungsten forms the complex $WO_2(C_{14}H_{12}O_2N)_2$, but quant. pptn. can be achieved only in the presence of quantities of KF too critical for unknown samples. The solubility of the tungsten complex in $CHCl_3$ is too low for quant. extraction; V^V forms an immediate yellow complex at pH 3. This is impure and changes on long standing into a white complex which has the composition $VO_2(C_{14}H_{12}O_2N)$. This is always contaminated with excess of reagent and is of no analytical value. The yellow complex is readily sol. in $CHCl_3$ and V^V can be extracted with benzoin α-oxime in $CHCl_3$ at pH 2·2 and determined colorimetrically with 8-hydroxyquinoline; Bi^{III}, U^{VI} and Sb^{III} interfere slightly. The Cr^{VI} complex with benzoin α-oxime was unstable and of no value for gravimetric or solvent-extraction procedures.

W. T. Carter

111. **Colour complexes of catechol with molybdate.** G. P. Haight, jun., and V. Paragamian (Swarthmore Coll., Pa., U.S.A.). *Anal. Chem.*, 1960, **32** (6), 642–644.—The reaction of catechol with Mo^{VI} in aq. systems was studied. Two complexes (2:1 and 1:1) are formed, the most suitable for analysis being the 2:1 complex, the formation of which is most complete in neutral soln., which is maintained by a $Na_2S_2O_5$ buffer; this also prevents oxidation of the catechol by air. The molar absorbances and formation constants for both complexes are given.

G. P. Cook

112. **The surface acidity of tungsten(VI) and molybdenum(VI) oxides.** M. L. Freedman (Refractory Metals Lab., General Electric Co., Cleveland, Ohio, U.S.A.). *Anal. Chem.*, 1960, **32** (6), 637–639.—Potentiometric titration with NaOH at high ionic strength was used. The surface acidity and solubility characteristics of tungsten trioxide depended on crystalline imperfections. Molybdenum trioxide did not exhibit surface acidity and the limiting pH for its continuous dissolution depended only on particle size. The layer lattice structure of molybdenum trioxide prevents the complete surface shielding which is postulated for tungsten trioxide.

D. C. Armsworth

113. **Analytical chemistry of uranium. I. The determination of uranium by luminescence.** V. F. Grigor'ev, V. F. Luk'yanov and E. P. Duderova. *Zhur. Anal. Khim.*, 1960, **15** (2), 184–190.—The method depends on the measurement of the luminescence of fused discs and beads, prepared under controlled conditions with a suitable flux (*e.g.*, NaF or fluoride - carbonate mixtures). The apparatus described includes an electric furnace with a detachable platinum case for preparing the beads, a tube with piston for proportioning the powdered flux, an apparatus for granulating the flux, and a photoelectric fluorimeter. A rapid method for preparing the beads is described. For a specimen with uniform distribution of U, the reproducibility of the readings is ±1·5%. For discs prepared with fluoride - carbonate flux and pure uranyl salts, the reproducibility is ±2%, and with NaF as flux, 6 to 7%. Methods for determining U in ores without preliminary purification are described.

R. E. E.

114. **Rapid extraction and spectrophotometric determination of uranium with 2-thenoyltrifluoroacetone.** S. M. Khopkar and A. K. De (Dept. of Chem., Jadavpur Univ., Calcutta, India). *Analyst*, 1960, **85**, 376–379.—To an aq. soln. of the sample (≡ 0·2 to 1·2 mg of U) is added 10 ml of an acetic acid - ammonium acetate buffer soln. (pH 6) and, if other cations (*e.g.*, Ag^+, Th^{4+}, Zr^{4+}, Cu^{2+} or Fe^{3+}) are present, 2 ml of 0·1 M EDTA (disodium salt), and the mixture is extracted with 10 ml of 2-thenoyltrifluoroacetone (**I**) (≃0·15 M) in benzene. The extinction of the yellow uranyl - **I** soln., appropriately diluted with benzene, is measured at 430 mμ against a reagent blank soln. treated similarly. At this wavelength the molar extinction coeff. (calculated on the basis of U) is 1954 ± 40. Oxalate, citrate, tartrate and CO_3^{2-} interfere, but 100-mg amounts of Pb^{2+}, Hg^{2+} or Ba^{2+} can be tolerated. As little as 2 μg of U per ml can be detected.

A. O. Jones

115. **Spectrophotometric determination of uranium with 1-(2-pyridylazo)-2-naphthol.** Shozo Shibata (Gov. Ind. Res. Inst., Nagoya, Japan). *Anal. Chim. Acta*, 1960, **22** (5), 479–484.—The ammoniacal test soln. containing 2 to 100 μg of U is treated with 1-(2-pyridylazo)-2-naphthol (**I**) in the presence of NaCl or Na_2SO_4 and the U-containing ppt. is extracted with $CHCl_3$. The sodium salts are necessary for complete extraction of the complex into $CHCl_3$. The extinction is measured at 560 mμ, with a reagent blank as standard. The spectra and the effect of pH on the complex are discussed. Beer's law is obeyed for 1 to 10 p.p.m. of U. EDTA competes with **I** and more **I** must be added when EDTA is present. The standard deviations of extinctions 0·180, 0·488 and 0·804 are, respectively, ±0·0021, 0·0040 and 0·0075 (11 determinations); thorium causes a positive error of <4% when in 40-fold excess. Of 24 metals listed, only Al and Sn interfere seriously; CN^- masks interference from Cu, Ni and Hg.

R. M. S. Hall

116. **Spectrophotometric determination of uranium with 3-(2-arsonophenylazo)-4,5-dihydroxynaphthalene-2,7-disulphonic acid (trisodium salt) [arsenazo].** H. P. Holcomb and J. H. Yoe (Pratt Trace Analysis Lab., Dept. of Chem., Univ. of Virginia, Charlottesville). *Anal. Chem.*, 1960, **32** (6), 612–617.—Uranyl ions give a stable blue complex with arsenazo, with an absorption max. at 590 mμ and a sensitivity of 1 in 3×10^7. The U is separated from interfering ions by extraction with ethyl ether by a modification of the method of Yoe *et al* (*Anal. Chem.*, 1953, **25**, 1200). An aliquot (5 ml) of the filtrate from this procedure is adjusted with HNO_3 to pH 1 to 2, boiled to remove CO_2, and then adjusted to a pH between 2·5 and 5·5. Arsenazo soln. ($2·1 \times 10^{-3}$ M) (2 ml) is added, followed by Clark and Lubs borate buffer (pH 8·6) (10 ml), the mixture is made up to 50 ml and the extinction is read at 596 mμ against a reagent blank and referred to a calibration curve. Beer's law is obeyed over

e range 0·5 to 10 p.p.m. of U, and the colour tensity is constant at temp. between 15° and 35°. ie coeff. of variation is 1·3%. R. E. E.

117. **Polarographic determination of sexavalent anium in the presence of excess of ferric iron.** A. Verbeek, J. T. Moelwyn-Hughes and E. T. erdier (Dept. of Chem., Univ. of Natal, Pieter-aritzberg, S. Africa). *Anal. Chim. Acta*, 1960, **22**, 570–576 (in English).—Uranyl ions at concn. of $^{-3}$ to 10^{-4} M are determined in the presence of a 400-fold excess of Fe^{3+}. The supporting ectrolyte is NaF soln. (≃0·75 M), but the concn. not critical; the pH must be >6·5. Titanium terferes, but Cu^{2+} and Mn^{2+} do not; Pb^{2+} in ncn. $>10^{-3}$ M should be removed to avoid pptn. th the electrolyte. The precision is within ±4% the 95% confidence level. R. M. S. Hall

118. **Determination of the stoicheiometry of anium dioxide. Polarographic determination of anium(VI) in uranium dioxide.** Hisashi Kubota nal. Chem. Div., Oak Ridge National Lab., Tenn., S.A.). *Anal. Chem.*, 1960, **32** (6), 610–612.—xavalent U in uranium dioxide can be determined dissolving the sample in hot H_3PO_4 in an inert mosphere and then polarographing in a H_3PO_4 - ClO_4 medium. The deviation from the stoicheio-etric composition of UO_2 is calculated by reference the O to U ratio of 2:1. The method is best plied to concn. of U in the range 5 to 50 μg per , and to samples that are nearly stoicheiometric; e composition of a sample having an O to U ratio 2·03:1 or less can be determined to within 0·001 atom. K. A. Proctor

119. **Determination of neptunium in uranium-sion-product mixtures. Initial extraction with butyl methyl ketone.** W. J. Maeck, G. L. Boo-an, M. C. Elliott and J. E. Rein (Atomic Energy v., Phillips Petroleum Co., Idaho Falls, U.S.A.). *eal. Chem.*, 1960, **32** (6), 605–607.—In the two-cle extraction system described, Np is oxidised th $KMnO_4$ and quant. extracted into isobutyl thyl ketone as a nitrate complex from an acid-ficient aluminium nitrate salting soln. containing rapropylammonium nitrate. By shaking with Cl_2 - hydroxyammonium chloride soln. the Np is ultaneously reduced and extracted from the ganic phase and is then re-extracted into thenoyl-fluoroacetone - xylene. The extraction of U and is <0·1 and <0·01%, respectively. Ruthenium d Zr are decontaminated by a factor greater than < 10^4. K. A. Proctor

120. **Rapid method for the determination of oride in liquids.** S. A. Bartkiewicz and J. W. binson (Esso Res. Lab., Esso Standard Oil Co., ton Rouge, La., U.S.A.). *Anal. Chim. Acta*, 60, **22** (5), 427–431.—The sample (15 g for 100 .m. of F^-) is introduced into a modified Beckman y-hydrogen burner connected to a Vicor furnace, l the products of combustion are scrubbed in a ttered glass scrubber containing glass beads. y conventional method can be used to determine in the scrubber, and the thorium nitrate pro-ture (Belcher *et al.*, *Anal. Abstr.*, 1954, **1**, 492) satisfactory. Tests with compounds that are rmally difficult to decompose gave recoveries of to 95%. The method is quick (20 min. per alysis) and any size of sample can be burned. examination of 1,2-dihydroxyanthraquinon-3-methylamine-*NN*-diacetic acid as a colorimetric agent for F^- showed results to be satisfactory for 0 to 10 p.p.m. of F^-, but Al, Fe, Ni, V, B and Cu interfere at the 100 p.p.m. level. R. M. S. Hall

121. **Determination of chloride by cathodic stripping polarography. Application to solutions of uranyl sulphate.** R. G. Ball, D. L. Manning and O. Menis (Oak Ridge National Lab., Tenn., U.S.A.). *Anal. Chem.*, 1960, **32** (6), 621–623.—In the method described, which is more sensitive than conventional polarographic methods, chloride in the concn. range 0·2 to 2 μg per ml is deposited as Hg_2Cl_2 on a mercury-pool micro-electrode at +0·4 V *vs.* the S.C.E. for 15 min., the sample soln. being stirred magnetically during the deposition. By changing the potential to less positive values the deposit is stripped from the electrode, and the peak height of the recorded current - voltage curve is measured and related to the chloride concn. by means of a calibration curve. The coeff. of variation is ≃5%. K. A. Proctor

122. **Argentimetric determination of chlorine in tetra-n-butyl titanate.** Ya. I. Chulkov. *Zavod. Lab.*, 1960, **26** (5), 550.—The sample (0·3 to 1 g) is dissolved, at 110° to 140°, in a mixture of 15 ml of acetic anhydride and ≮4 g of NH_4F. To dissolve the ppt., the soln. is heated at 50° to 55° with the minimum amount of water. The Cl^- in the cooled soln. are then determined by titration with $AgNO_3$ soln. G. S. Smith

123. **The adsorption of some chloro-complexes on cellulose.** T. J. Beckmann and M. Lederer (Inst. du Radium, Lab. Curie, Paris, France). *J. Chromatography*, 1960, **3** (5), 498–499 (in French).—Un-impregnated cellulosic paper has been shown to adsorb chloro and bromo anionic complexes from solutions in HCl or LiCl. Results for the chromatography of anions from Au^{III}, Ce^{III} and Fe^{III} in solutions of HCl and of LiCl containing 1% of 10 N HCl, by an ascending technique, are presented and show possible value for analysis. S. M. Marsh

124. **The simultaneous determination of bromide and iodide.** A. de Sousa (Anal. Chem. Lab., Fac. Sci., Lisbon, Portugal). *Anal. Chim. Acta*, 1960, **22** (6), 520–522 (in French).—Two aliquots of sample soln. are treated with $AgNO_3$. The ppt. from one aliquot is oxidised with $K_2Cr_2O_7$ and H_2SO_4 to convert I^- into IO_3^- and liberate Br, which is removed in a current of air. The IO_3^- are then reduced to I^- with Na_2SO_3 and pptd. as AgI. This ppt. and the ppt. of combined halides from the second aliquot are dissolved in ammoniacal nickel tetracyanide, and the displaced Ni^{2+} are titrated with EDTA (disodium salt). Bromide is determined by difference. R. M. S. Hall

125. **Complexometric determination of bromides and bromates in the presence of each other.** A. de Sousa (Chem. Lab., Fac. Sci., Univ. of Lisbon, Portugal). *Z. anal. Chem.*, 1960, **174** (5), 337–339 (in English).—The method described is based on that of Flaschka and Huditz (*Brit. Abstr. C*, 1953, 51). Actual bromide is pptd. as AgBr, which is dissolved in an ammoniacal soln. of $K_2Ni(CN)_4$, and the liberated Ni^{2+} are titrated with EDTA (disodium salt) soln., with murexide as indicator. Bromate, in another portion of sample, is reduced to bromide with arsenous oxide, and the total bromide is determined as described above. Chlorides, iodides and heavy metals interfere. W. T. Carter

126. **Coulometric determination of small amounts of iodide by internal electrolysis.** J. Kis and C. Schejtanow (Inst. f. Anorg. Chem., Tech. Univ., Budapest). *Period. Polytech.*, 1960, **4** (2), 163–170 (in German).—The determination of 30 to 100 μg of iodide can be carried out in $\simeq$30 min. by internal electrolysis, with the hydrogen micro-coulometer described by Proszt and Poós (*Ibid.*, 1957, **1**, 25) to measure the amount of electricity which flows during the oxidation of iodide to iodine. The cathode consists of a lead dioxide - sulphuric acid paste contained in an extraction thimble impregnated with collodion; connection to it is made by a carbon rod. The anode is a cylinder of platinum gauze surrounding the cathode, and connected to it through the micro-coulometer. The basal soln. (2% aq. KNO_3) is stirred by a stream of N. The relative error of the determination is $<3\%$.

R. M. Rowley

127. **Photometric determination of manganese.** H. Hartkamp (Inst. f. Spektrochem. u. angew. Spektroskopie, Dortmund-Aplerbeck, Germany). *Angew. Chem.*, 1960, **72** (10), 349.—Pyridine-2,4-dicarboxaldoxime and pyridine-2,6-diacetaldoxime (**I**) at pH 9·5 to 11·5 are suitable for the photometric determination of Mn in the range 0·1 to 10 μg per ml. Soluble green complexes, probably containing Mn^{III}, and becoming red at high metal concn., are formed with Mn^{2+}, and obey Beer's law over the concn. range stated. The colour develops very rapidly after warming the test soln. for a short while with a small excess of reagent, and does not alter for several hours. Reducing agents, *e.g.*, ascorbic acid, hydroxyammonium chloride and hydrazine sulphate, interfere. Metals of groups I to IV are without influence, but Fe, Co and Ni must be absent. The complex with **I** has a max. extinction at 598 mμ.

J. H. Waton

128. **Mass-spectrometric identification and determination of technetium.** G. M. Kukavadze, R. N. Ivanov, V. P. Meshcheryakov, Yu. G. Sevast'yanov, B. S. Kir'yanov, V. I. Galkov and A. P. Smirnov-Averin. *Atomnaya Energiya*, 1960, **8** (4), 365–366.—Surface ionisation of the test substances on incandescent iridium-coated (0·1 μ) tungsten emitters provides the ion stream. Aq. ammonium pertechnetate soln. is transferred by micro-pipette to the emitter. Technetium is then reduced to the metallic state by heating the emitter to redness in a stream of H for a few minutes. The ion source with the treated sample is placed in the mass spectrometer, and Tc^+ are produced at 1600° to 1800°. Mass spectrograms of ^{99}Tc and of ^{87}Rb and ^{85}Rb are given; Rb provides a reference peak, and is present as an impurity in the iridium. The method described, in combination with isotope dilution or integral mass-spectrography, is satisfactory for the determination of Tc.

K. R. C.

129. **Method for "ferrous iron" and "excess oxygen" in rocks, minerals and oxides.** C. O. Ingamells (Mineral Constitution Lab., Pennsylvania State Univ., University Park, U.S.A.). *Talanta*, 1960, **4** (4), 268–273 (in English).—The method depends on the stability of both Mn^{III} and Mn^{II} in phosphoric acid - pyrophosphate mixtures. The sample is dissolved in a phosphoric acid mixture containing an excess of Mn^{II} with or without the addition of standard oxidant, and the Mn^{III} remaining in the soln., or produced by reaction with the sample, is titrated with $(NH_4)_2SO_4.FeSO_4$. *Procedure*—Mix 85% H_3PO_4 (20 ml), 50%$NaH_2PO_4.H_2O$ soln. (5 ml) and 0·1 N $K_2Cr_2O_7$ (0·5 ml) and heat without boiling until water is expelled and the excess of chromate is destroyed. Cool, add water (2 ml), 50% $MnSO_4.H_2O$ soln. (1 ml) and a measured excess of 0·1 N $KMnO_4$, evaporate off the excess of water and add the finely ground sample (>1 g). Heat until dissolution is complete, cool, dilute with water until the concn. of H_3PO_4 in the soln. is $\simeq$20% and titrate with 0·01 N $(NH_4)_2SO_4.FeSO_4$, with Ba diphenylaminesulphonate as indicator. The results obtained agree well with those obtained by other methods, but the procedure is not applicable to samples that do not dissolve readily in the reagent, or that form peroxides in acid soln. or that contain sulphur or organic carbon; Co_3O_4, Pb_3O_4 and certain rare-earth-metal oxides evolve oxygen during dissolution and give low results for excess oxygen.

W. T. Carter

130. **Analytical aspects of some organic acids. VI. Direct complexometric titration of iron(III) and indirect EDTA titration of zirconium and thorium using *o*- and *p*-cresotic [2- and 6-hydroxy-*m*-toluic] acids as indicators.** C. S. Pande and T. S. Srivastava (Lucknow Univ., India). *Z. anal. Chem.*, 1960, **175** (1), 29–34 (in English).—Macro (3 to 7·8 mg) and micro (0·16 to 2·6 mg) amounts of Fe^{3+} can be titrated directly with EDTA, with 2- or 6-hydroxy-*m*-toluic acid (**I**) as indicator. At the end-point the colour change is from blue-violet to pale yellow. *Procedure*—The test soln. containing 0·16 to 2·6 mg of Fe^{3+} is adjusted to pH 3·6 by adding acetate buffer and diluting with twice-distilled H_2O to 25 ml. After an addition of 0·05 g of solid **I** the soln. is titrated with 0·05 M EDTA (disodium salt) until the violet colour is discharged. There is no interference from Be, Mg, Ca, Sr, Ba, Cd, UO_2^{2+} or Mn^{2+}, but Cu^{2+}, Zn^{2+}, Pb^{2+}, Fe^{2+}, Co^{2+}, Ni^{2+}, ZrO^{2+}, Al^{3+}, Cr^{3+}, Ti^{4+}, Ce^{4+} and Th^{4+} should be absent. The indirect EDTA titrations of Zr and Th, with **I** as indicator, have also been carried out. To the sample soln. is added 0·1 M EDTA (disodium salt) in slight excess, and the pH is adjusted to 6·0 by adding dil. aq. NH_3 and boiling the soln. gently for $\simeq$2 min. The soln. is then cooled to 31°, the pH is adjusted to 4 or 3·6 and the soln. is diluted to $\simeq$100 ml. Excess of EDTA is back-titrated with 0·1 M Fe^{3+}, with 0·1 g of **I** as indicator. Some typical results are given.

B. B. Bauminger

131. **Low-pressure method for carbon analysis in steels: a note.** V. Ramakrishna (Research & Control Lab., Tata Iron & Steel Co., Jamshedpur, India). *Anal. Chim. Acta*, 1960, **22** (6), 592–594 (in English).—The carbon dioxide cold-trap of Cook and Speight (*Analyst*, 1956, **81**, 144), when used with liquid-air coolant, froze out oxygen from the gas stream. A modified trap is proposed and described together with a method for its use.

R. M. S. Hall

132. **Determination of tin in pig iron and steel.** C. Mahr and K. Waffenschmidt. *Arch. Eisenhüttenw.*, 1960, **31** (4), 221–225.—Four procedures are given for the determination of about 0·005 to 0·1% of Sn. Three of these, suitable only for samples containing $<2\%$ of Cr, rely on co-pptn. of the hydroxides of Sn and Al, or distillation of $SnCl_4$, or a combination of both techniques, followed by polarographic determination in a basal soln. 10^{-4} M in tetraphenylarsonium chloride and 6 M in HCl containing a few drops of cresol red. In the fourth procedure, the sample is dissolved in an oxidising

dium, an excess of iron powder is added, the xture is heated under a Bunsen valve, and the uced sample is titrated potentiometrically with O_3 soln. (0·005 N) after addition of KI.
T. R. Andrew

133. **Determination of nitrogen in steel.** J. Llop p (Lab. de Altos Hornos de Cataluña, S.A.). *t. Hierro y Acero*, 1960, **13**, 532–535.—Some rogen is lost when steel is dissolved in HCl but when H_2SO_4 is used. The sample of carbon or y steel (3·5 g) is dissolved in H_2SO_4 (1:4) ml) in an atmosphere free from NH_3. After addition of tartaric acid, the soln. is transferred a steam-distillation apparatus (illustrated) and de alkaline. The NH_3 is distilled off and orbed in 0·005 N H_2SO_4, the excess of acid being k-titrated with 0·025 N NaOH. A reagent nk is carried out, and the content of nitride rogen is calculated. Nitrides not decomposed dil. H_2SO_4 are filtered off, fused with K_2SO_4 and led to the sample soln. The procedure takes 20 n. G. H. Foxley

134. **The accurate determination of cobalt.** A. G. ster and W. J. Williams (Royal Holloway College, glefield Green, Surrey, England). *Anal. Chim. a*, 1960, **22** (6), 538–546 (in English).—A critical iew is given of the possible primary standards the determination of Co, and, of those investi- ed, $[Co(NH_3)_5Cl]Cl_2$, $[Co(NH_3)_6]Cl_3$ and $[Co(CN)_6]$ are preferred. The errors are <0·7%. R. M. S. Hall

135. **Complexometric estimation of micro amounts cobalt using ethylenedinitrilotetra-acetic acid.** N. Sen Sarma (Chem. Engng Dept., Jadavpur iv., Calcutta, India). *Anal. Chem.*, 1960, **32** 717–719.—The method depends on the titration th EDTA (disodium salt) of the blue soln. formed en acetone and an excess of NH_4SCN are added a neutral or acetic acid soln. of Co^{II}. The end- nt (colourless or pink) may be determined ually or photometrically. As little as 2·5 μg of per ml may be determined visually, or 1 μg per photometrically. Pyrophosphate, citrate, oxa- e and cations other than alkali metals interfere; derate amounts of orthophosphate, fluoride l tartrate can be tolerated. Modified procedures described for the determination in the presence alkaline-earth metals, Fe, Al, Cu and Ni. The t results are obtained when the soln. for titration tains 5% and 30 to 35%, respectively, of NH_4SCN and acetone, and <5 μg (visual) or 4 μg otometric) of Co per ml. R. E. E.

136. **Determination of cadmium as impurity in kel and cobalt by ion exchange.** V. N. Pavlova l N. P. Strel'nikova. *Zavod. Lab.*, 1960, **26** 536–537.—Cadmium is adsorbed on a column DÉ-10p from a soln. in 2 N HCl, whilst Ni, Co, and Fe are not adsorbed; Cd is eluted with cer and determined colorimetrically with dithi- e. G. S. Smith

137. **Determination of ruthenium in the presence uranium by dithio-oxamide.** V. I. Shlenskaya. *tn. Moskov. Univ., Ser. Khim.*, 1960, II, No. 2, 72.—The method is based on the formation of a e complex by ruthenium salts in aq. ethanolic l soln. with dithio-oxamide (**I**). If the ratio of to U is ≃1:100, adopt the following procedure. d conc. HCl (1 ml) and 0·1% ethanolic **I** soln. the ruthenium salt soln. and heat it on a boiling-water bath for 30 min. Make the soln. up to 25 ml with ethanol and measure the extinction at 610 to 660 mμ against an ethanolic **I** soln. (2 ml of 0·1% ethanolic **I** soln. per 25 ml of ethanol) as the blank. Beer's law is obeyed in the range 10 to 60 μg of Ru per 25 ml of soln. With $\lambda_{max.} = 650$ ($\epsilon_{max.} = 8500$) the sensitivity is 0·23 μg of Ru per ml. If the ratio of Ru to U is >1:100, adopt the following procedure. Add conc. HCl (1 ml) and 0·1% ethanolic **I** soln. (1 to 2 ml) to a soln. containing 10 to 50 μg of Ru. Heat the soln. for 5 min. on a boiling-water bath, allow it to cool and carry out a triple extraction with isoamyl alcohol (5 + 5 + 5 ml). Make the combined extracts up to 25 ml with isoamyl alcohol. Measure the extinction at 610 to 660 mμ against isoamyl alcohol as the blank soln. Beer's law is again obeyed for the same concn. range. K. R. C.

138. **Absorptiometric determination of palladium as its 2-mercapto-4,5-dimethylthiazole complex.** A. J. Radford (Geological Survey Office, Dodoma, Tanganyika). *Analyst*, 1960, **85**, 445–448.—Ryan (*Ibid.*, 1951, **76**, 310; 1950, **75**, 557) states that the determination of Pd with 2-mercapto-4,5-dimethylthiazole is not affected by changes in acidity and concn. of NaCl. The method was studied in detail and the absorption measurements were extended into the u.v. region. When the complex is measured in the u.v. region with a Spekker absorptiometer and a mercury-vapour lamp there is an increase of 160% in sensitivity and the soln. obeys Beer's law. The acidity must be controlled when measurements are made in the u.v. region, but need not be when a tungsten-filament lamp is used. Addition of ethanol to prevent turbidity is not necessary. Nitric acid oxidises the reagent and must be removed. A. O. Jones

139. **New gravimetric methods for the micro-determination of osmium.** P. Spacu and C. Gheorghiu (Inorg. Chem. Lab., Univ. "C.I. Parhon," Bucharest, Romania). *Z. anal. Chem.*, 1960, **174** (5), 340–345 (in German).—Three weighing forms for osmium are described—(*a*) $[OsCl_6]H_2.2NH_2C_6H_4CH_2C_6H_5$ is pptd. from HCl soln. containing $[OsCl_6]^{2-}$ by acridine soln. (2% in acetic acid) and dried *in vacuo* after being washed with ethanol and ethyl ether; (*b*) $[OsBr_6]H_2.2NH_2C_6H_4CH_2C_6H_5$ is similarly pptd. from HBr soln. containing $[OsBr_6]^{2-}$, and can be dried at 110°; (*c*) $[Os(NH_2CSNH_2)_6][Cr(SCN)_6]$ is pptd. from H_2SO_4 or HCl soln. containing OsO_4 by acid thiourea soln. followed by $K_3[Cr(SCN)_6]$ soln. and can be dried at 105°. The preparation of $K_3[Cr(SCN)_6]$ is described. The pptn. of (*a*) and (*b*) can be carried out in the presence of the alkali metals, the alkaline-earth metals, Cu^{2+}, Fe^{2+}, Cr^{3+} and Co^{2+}; form (*c*) is used with soln. obtained by the distillation of OsO_4. All three pptn. are accurate to within ≃±2% for amounts of Os in the range 0·5 to 5 mg.
W. T. Carter

140. **Coulometric titration with electro-generated tin(II). Titration of platinum(IV).** A. J. Bard (Univ. Texas, Austin, U.S.A.). *Anal. Chem.*, 1960, **32** (6), 623–625.—Quadrivalent Pt is reduced only to Pt^{II} by an equivalent amount of Sn^{II} in an acid bromide medium. A slight excess of Sn^{II} is generated and back-titrated with electro-generated Br, with potentiometric or spectrophotometric end-point detection; 2 to 13 mg of Pt can be titrated with an average error of ±0·5% with

potentiometric end-point detection. Osmium, Ru, Cu, Fe and Au interfere and the Pt must then be separated; Pd^{II}, Rh^{III} and Ir^{IV} interfere only slightly. (*Cf.* Bard and Lingane, *Anal. Abstr.*, 1960, **7**, 511, 889.) D. C. ARMSWORTH

141. **Assay procedure for platinum in reforming catalysts.** I. Maziekien, L. Ermanis and T. J. Walsh (Engelhard Industries, Inc., Newark, N.J., U.S.A.). *Anal. Chem.*, 1960, **32** (6), 645–647.—The extinction of the Pt - $SnCl_2$ complex is measured. Fresh catalyst (3 g) is dissolved in HCl (1:1) (150 ml) by heating and the soln. is filtered into a 2-litre flask; $AlCl_3$ soln. (45·4% aq.) (100 ml), conc. HCl (650 ml) and $SnCl_2$ soln. [28% in HCl (1:1)] (60 ml) and water are added, the soln. is mixed and made up to vol. The extinction is read in a Corex cell in a recording spectrophotometer at 403 mμ, scanning from 375 to 425 mμ. Standard Pt soln. are measured alternately with sample soln. to provide the conversion factor. Spent catalyst is ignited to remove C and S, then dissolved in aqua regia, and the Pt is separated with H_2S or by reduction to metal with granulated zinc. The ppt. is dissolved in aqua regia, the HNO_3 is removed by evaporation, and the soln. is treated as described above. An alternative separation with zinc for use when large amounts of Fe or Mo are present, is also described. Full experimental details are given. R. E. E.

142. **The heterometric determination of platinum, palladium and gold by titration with 1,10-phenanthroline.** M. Bobtelsky and M. M. Cohen (Dept. of Inorg. and Anal. Chem., Hebrew Univ., Jerusalem). *Anal. Chim. Acta*, 1960, **22** (6), 532–538 (in English).—Instructions with experimental details are given for the determination of Pt^{IV}, Pd^{II} and Au^{III} as chlorides in thiocyanate or chloride soln. with 1,10-phenanthroline at pH 1 to 7. The procedures for the analysis of binary mixtures of these ions and also for the determination of 1,10-phenanthroline with $PdCl_2$ are described. The error, in most cases, is zero. R. M. S. HALL

143. **Measurement of thin metal layers. Fluorescent X-ray production by radio-isotope sources.** G. B. Cook, C. E. Mellish and J. A. Payne (Isotope Res. Div., A.E.R.E., Harwell, England). *Anal. Chem.*, 1960, **32** (6), 590–593.—By choosing the correct method (either *K* X-ray excitation, *L* X-ray excitation or base-metal *K* X-ray absorption) and the correct source, it is usually possible to make the measurements sensitive at the thicknesses that are of interest. This flexibility is limited, however, by the absorptive properties of the metal under investigation. Measurements on chromium, copper and tin plate are reported and discussed. K. A. PROCTOR

144. **Rapid automatic X-ray fluorescence analysis of mineral powders and solutions from the hydrometallurgical works of the Union Minière du Haut-Katanga.** A. Hans (C.N.R.M., Section de Liège). *Chim. Anal.*, 1960, **42** (5), 236–244; (6) 282–286.—Copper, zinc and cobalt have been determined in cuprozinc and cuprocobalt minerals and concentrates. A tungsten or gold anti-cathode is used with voltages from 25 to 30 kV. The K_α lines of the elements are used for the determinations. The sample (2 g) is dissolved in acid and used as a liquid soln., or the powdered sample (0·2 g) is mixed with BaO_2 (2 g) and fused with borax (10 g) and used as a "solid" soln. By such means, absorption effects are eliminated, and an internal standard is unnecessary. The calibration curves for the soln. method show slight curvature but not those for the borax-bead method. The ranges of concn. covered are—Zn, 0·05 to 60%; Cu, 0·05 to 50%; and Co, 0·05 to 5%. The method, suitable for routine process control, for the determination of Co (1·5 to 3%) and Cu (0·5 to 5%) in copper electrolysis soln. containing 0·5 to 5% of free H_2SO_4, involves irradiation with X-rays (Co K_α and Cu K_α, respectively) and measurement of the resulting fluorescence with a scintillation counter. The concn. of Cu has much less influence on the fluorescence of Co than has Co on that of Cu. To minimise these effects the soln. are diluted (1:10) and the free acid concn. is kept constant. The Co and Cu concn. can be rapidly determined with high precision by successive approximations to calibration curves. R. M. S. HALL
J. P. STERN

145. **Spectrochemical rubidium - strontium method for geological age determination.** H.-J. Eichhoff (Inst. for Inorg. and Nuclear Chem., Johannes Gutenberg Univ., Mainz, Germany). *Appl. Spectroscopy*, 1960, **14** (3), 74–76.—A flame method is described for the determination of Rb and Sr in lepidolites after preliminary ion-exchange separations and with In as an internal standard. The abundance of ^{87}Sr is determined by excitation of the mineral in a hollow-cathode source, and the examination of the hyper-fine structure of Sr 4078 A, obtained by means of a Fabry-Perot etalon. Results of geological age determinations made by this method are in good agreement with those obtained by other methods and with existing geological theories. P. T. BEALE

146. **Gravimetric method for the determination of barium oxide in Portland cement.** C. L. Ford (A.S.T.M., 1916 Race Street, Philadelphia, Pa., U.S.A.). *Bull. A.S.T.M.*, 1960, (247), 77–80.—By the procedure given, from 0·01 to 0·20% of BaO may be determined with a max. error of 0·015%. The sample (1 g) is dissolved in hot dil. HCl, the soln. is diluted to $\simeq$250 ml (to prevent subsequent pptn. of $CaSO_4$), then boiled, and $BaSO_4$ is pptd. by the addition of H_2SO_4 (1:1). The mixture is kept at $\simeq$100° for 12 to 16 hr., then filtered, and the ppt. is ignited at 800° to 900° for 15 min.; HF is then added to decompose the HCl-insol. matter and volatilise any SiO_2. The residue is fused with $K_2S_2O_7$, the melt is extracted with H_2O (the $BaSO_4$ remains insol.), H_2SO_4 (1:1) is again added and, after 16 to 18 hr., the hot soln. is filtered through Whatman No. 42 paper. The $BaSO_4$ is washed, dried and ignited to const. wt. at 800° to 900°. The accuracy of the method was verified by analyses of mixtures containing known amounts of Ba. W. J. BAKER

147. **Quantitative analysis of radionuclides in process and environmental samples.** A. L. Bond (Savannah River Plant, E.I. du Pont de Nemours & Co., Inc., Aiken, S.C., U.S.A.). *Anal. Chem.* 1960, **32** (6), 599–604.—An ion-exchange procedure combined in some cases with radiochemical pptn. has been developed for the separation and recovery of Ru, Zr, Nb, Np, Co, Fe, Zn, Sr, rare-earth elements, Cr and Cs from natural materials. Iodine is eliminated by volatilisation. The recovery of individual isotopes is >95%, with a standard

iation of ±4% (P = 0·9). The decontamination tors are > 10^4 for all ion-exchange separations . > 10^3 for gravimetric separations.

K. A. Proctor

ee also Abstracts—**153**, Determination of Hg. , Separation of O, N, CO_2 and N_2O by molecular re. **309**, Heavy metals in sugar syrups. **337**, Cu l Fe in water. **341**, Fission products in water. , Chromatography of oxidisable metals in an rt atmosphere.

3.—ORGANIC ANALYSIS

Determination of elements and radicals and of organic compounds not included in other sections. Organic industrial products, including petroleum and its products, fuels, detergents, volatile oils, cosmetics, dyestuffs, fibres, plastics, resins, paints, elastomers, leather, explosives, etc.

48. **Interaction of beta-particles with matter. antitative determination of hydrogen, carbon, rogen, oxygen and fluorine materials.** P. R. ay, D. H. Clarey and W. H. Beamer (Radiochem. ., The Dow Chemical Co., Midland, Mich., .S.A.). *Anal. Chem.*, 1960, **32** (6), 582–589.—The of β-ray back-scattering and β-ray transmission the analysis of liquids is accurate, rapid, simple l non-destructive. Binary compounds, such as drocarbons, can be completely analysed by ner method, whereas ternary compounds require nsity measurements by both methods for a nplete quant. analysis. Compounds and mix- es containing more than three elements can only analysed if quant. data are available for all the ments in excess of three. The standard deviation the experimentally determined weight fraction of in hydrocarbons and ternary compounds is ·03%. The precision is approximately the same both the back-scatter and transmission instru- nts. K. A. Proctor

49. **Spectrographic method of determining traces metallic] impurities in organic compounds.** Sh. I. zulaev, L. K. Popova and R. L. Slyusareva. *od. Lab.*, 1960, **26** (5), 552–553.—The sample) is mixed with 315 mg of spectrographically e $SrSO_4$ and evaporated to dryness, the residue reated with 0·5 ml of conc. H_2SO_4, the soln. is porated to fuming, and the residue is ignited . powdered. With difficultly volatile compounds sample is mixed with $SrSO_4$ and 2 ml of conc. SO_4 and evaporated nearly to dryness, followed treatment with H_2SO_4 and HNO_3, evaporation, The spectrum is excited in a d.c. arc between on electrodes, and 22 elements are determined means of standards on a base of $SrSO_4$.

G. S. Smith

50. **Analysis of ternary solvent mixtures.** F. me (Inst. Entwicklung chem.-phys. Analysen- thoden, Weilheim/Obb). *ChemikerZtg*, 1960, (9), 291–294.—Since the determination of n nponents requires the measurement of $(n - 1)$ ameters, the measurement of the dielectric stant must be supplemented for such cases. er suggested measurements are those of re- tive index and density and a further extension he measurement of water content by the Karl her method before the determination of a single sical constant. A system of triangular co-ordinates and the resolution of a mixture by progressive separation of the components with a molecular sieve are described. E. G. Cummins

151. **Apparatus for hydrogen determination in hydrocarbons using β-radiation.** M. Přibyl (Military Acad., Brno, Czechoslovakia). *Chem. Průmysl*, 1960, **10** (5), 240–243.—The procedure previously described (*Anal. Abstr.*, 1960, **7**, 3785) is modified. The apparatus (illustrated) comprises a radiation source (20 mC of ^{90}Sr), a measuring and a reference ionisation chamber, and a measuring and an auxiliary wedge filter. The difference in the ionisation currents is measured by means of a recording galvanometer. The determination takes 20 min., and has an accuracy of ±0·03%. Small amounts of O, N and S do not interfere, but corrections must be applied for larger amounts. Calibration is carried out with pure hydrocarbons. The apparatus can be adapted for continuous measurement, and the method is suitable for analysis of fuels and lubricating oils. J. Zýka

152. **Determination of sulphur in organic compounds with metallic silver as absorbent. A critical examination.** J. P. Dixon (Shell Research Ltd., Thornton Res. Centre, Chester, England). *Talanta*, 1960, **4** (4), 221–226 (in English).—The sample is burnt by the "empty tube" method (*cf.* Ingram, *Anal. Abstr.*, 1956, **3**, 1745), the sulphur oxides produced are absorbed on electrolytically prepared silver and the Ag^+ equivalent to the S are extracted with boiling water and determined by potentiometric titration with 0·1 N KI, with glass and silver electrodes. In order to obtain blanks low enough for the determination of micro amounts of S, it is necessary to use boiled de-ionised water for the extraction, to wash the silver with ethanol and ethyl ether between analyses and to blanket it with nitrogen during cooling. With a 25-mg sample, 10 to 15 μg of S can be determined with a reproducibility of ≃3%, but Cl interferes seriously and must be absent. W. T. Carter

153. **Determination of mercury in organomercury compounds and certain inorganic compounds.** F. Martin and A. Floret (Soc. des Usines Chim., Rhône-Poulenc, Vitry-sur-Seine). *Bull. Soc. Chim. France*, 1960, (4), 610–613.—Two procedures are described. In the first the compound is placed between layers of CaS and heated in a slow stream of air. The vapours are passed through successive layers of granular CaO and silvered pumice (prep. described) at 750° to 800° and the Hg is absorbed in a tube containing gold leaf. The method is suitable for 1 to 20 mg of Hg. In the second, more generally applicable, procedure, the sample is pyrolysed in a stream of H and burnt in an oxy-hydrogen flame as previously described (*Anal. Abstr.*, 1960, **7**, 559), with the modification that a portion of the O feeding the flame is bubbled through satd. bromine water. The $HgBr_2$ so formed is condensed with the water and received in a beaker containing 10 ml of water. The excess of Br in the condensate is removed by adding hydrazine hydrochloride, in small portions, till the colour is discharged, and 0·1 g in excess. After stirring for 2 min., 0·1 M EDTA (disodium salt) (10 ml) is added, followed by a vol. of pyridine equal to the total vol. of the soln., and the Hg is determined by potentiometric titration with 0·01 M Na diethyldithiocarbamate. Results by the two methods range from 98 to 101% of the theoretical.

R. E. E.

154. **Gasometric determinations of certain organic nitrogen functions, and, more generally, of compounds capable of the quantitative liberation of a volume of gas.** L. Nicolas and J. Mansel (Compagnie Franç. des Matières Colorantes, Usine d'Oissel, France). *Chim. Anal.*, 1960, **42** (4), 171–180; (5), 226–235.—An apparatus is described which permits N to be determined in a wide range of organic materials. An extension is provided above the nitrometer, so that the N evolved is measured in a graduated tube over water. The CO_2 used to expel air from the apparatus is obtained by heating $NaHCO_3$. The sample, dissolved in cooled H_2O and, if necessary, HCl or acetone, is kept cold in an ice bath before addition to the reaction vessel which contains the boiling decomposition reagents. For the determination of N in diazo compounds (*e.g.*, aliphatic and aromatic diazo compounds, stabilised diazonium salts, diazotates and triazenes), the sample (0·2 to 1 g) in aq. soln. is added to 150 ml of H_2SO_4 (30%) containing 1 g of Cu_2Cl_2. Excellent agreement is obtained with theoretical results, except in those few cases when another gas is evolved besides N. When this gas is identified and dissolved in a suitable absorbent (*e.g.*, hydrocarbons in ethanol), then the correct result is given. Procedures are also described for the determination of N in diazosulphonates and phenylhydrazine derivatives. Compounds containing the $-NH_2$ group (*e.g.*, *p*-nitroaniline) are determined after diazotisation, aromatic compounds containing the –OH and $-NH_2$ groups by coupling with a known excess of a diazonium salt and determination of the excess, and compounds containing carbonyl groups (*e.g.*, aldehydes and ketones) by conversion into phenylhydrazones with a known excess of a phenylhydrazine derivative, and determination of the excess. A literature survey is given, with 99 references. J. H. Waton
R. E. E.

155. **Dinitrophenylation method for the microdetermination of amide nitrogen [in wool].** J. H. Bradbury (Div. of Textile Ind., Wool Res. Lab., C.S.I.R.O., Geelong, Australia). *Anal. Chim. Acta*, 1960, **22** (5), 444–448.—The procedure described is based on the reaction of NH_3 (derived from the protein by HCl hydrolysis) with 1-fluoro-2,4-dinitrobenzene to give 2,4-dinitroaniline (**I**), which is determined spectrophotometrically at 345 mμ after separation by paper chromatography. The yield of **I** is constant at 80% under the given conditions. The minimum amount of NH_3 necessary is $\simeq 2$ μg. With 0·5-mg wool samples the mean value was $7{\cdot}4 \pm 0{\cdot}4$ moles per 10^4 g of material (at least 4 determinations). Cuticle-rich samples may differ significantly from whole-fibre samples.
R. M. S. Hall

156. **Quantitative analysis of monochloride mixtures by means of infra-red absorption spectra.** V. I. Kolbasov, S. B. Bardenshteĭn and R. V. Dzhagatspanyan. *Zavod. Lab.*, 1960, **26** (5), 587–590.—The mixture of chlorides obtained by the chlorination of propylene is analysed by means of the absorption bands at 1875 cm^{-1} (allyl chloride), 880 cm^{-1} (2-chloropropene), 1319 cm^{-1} (*cis*-1-chloropropene), 855 cm^{-1} (n-propyl chloride) and 1060 cm^{-1} (isopropyl chloride). G. S. Smith

157. **Micro-determination of ethanol in aqueous media.** L. Bessot and M. Scemama (Lab. Phys., Fac. de Pharm., Paris, France). *Compt. Rend.*, 1960, **250** (23), 3897–3899.—The method of Nicloux is modified to avoid the preliminary distillation and the final titration with $Cr_2O_7^{2-}$. *Procedure*—Treat the sample with water (1 ml) and H_2SO_4 (1 ml) and heat to 85° for 3 hr. in a closed system that contains in a separate vessel 5 ml of 0·1 N $K_2Cr_2O_7$ in H_2SO_4 (1:1). The ethanol diffuses quantitatively into and reacts with the $K_2Cr_2O_7$. Determine the amount consumed, absorptiometrically, at 580 mμ. The error for ethanol (1·5 to 3·2 mg) is $< \pm 3\%$. J. P. Stern

158. **Studies on fermentation products. I. Fusel oil analysis by gas chromatography.** Akira Kamibayashi, Makoto Miki and Hideo Ono. *Rep. Ferment. Res. Inst., Chiba*, 1960, (18), 101–110 (in Japanese).—The samples (0·1 to 0·3 ml), containing n-butanol as internal standard, were analysed in a Perkin-Elmer Vapour Fractometer, model 154-C, with thermal conductivity detection and He as carrier gas. Three columns (4 mm × 2 metres) were used, containing the following stationary phases on a support of diatomaceous earth, *viz*, (*A*) tetraethylene glycol dimethyl ether, (*B*) polyoxyethylene glycol 1500 and (*C*) dibenzyl ether. The operating temp., inlet pressures and flow-rates were, respectively, (*A*) 74°, 30 p.s.i. and 112·4 ml per min., (*B*) 100°, 15 p.s.i. and 76·4 ml per min. and (*C*) 78°, 15 p.s.i. and 83·7 ml per min. Alcohols were identified in the peak fractions as 3,5-dinitrobenzoates (ethanol, n-propanol and iso-butyl alcohol), as the 3-nitrophthalate (isoamyl alcohol) or by optical activity (2-methylbutan-1-ol); isoamyl alcohol and 2-methylbutan-1-ol could not be separated on either column. Regression equations of the form $Y = aX + b$ were calculated, where Y is the ratio of wt. of alcohol to wt. of n-butanol, X is the ratio of the corresponding peak areas, and a and b are constants. Retention volumes relative to n-butanol on the three columns are tabulated for water and 11 alcohols. [From English summary.] R. E. E.

159. **Analysis of 1,2-glycols and polyhydric compounds. III. Direct iodimetric determination of glucose via the aldehyde formed by periodate oxidation.** L. Maros and E. Schulek. *Magyar Kém. Foly.*, 1960, **66** (4), 147–149.—This is a version (in Hungarian) of the article published (in German) in *Acta Chim. Acad. Sci. Hung.*, 1959, **21**, 91; *cf. Anal. Abstr.*, 1960, **7**, 3307.

160. **Microbiological assay for the quantitative determination of glycerol.** L. Eidus, B. B. Diena, A. C. Maniar and L. Greenberg (Biologics Control Lab., Dept. of Nat. Health and Welfare, Ottawa, Canada). *Canad. J. Microbiol.*, 1960, **6** (3), 283–287.—The cylinder-plate method described is based on the observed inhibitory effect of glycerol on *Staphylococcus aureus*. Soln. of glycerol in the range of 20 to 80 mg per ml are incubated for 16 to 18 hr. on a previously seeded medium (prep described), and the amount corresponding to zone size is read from a standard curve. Variation of results from a known concn. was $\simeq 12\%$, as compared with $\simeq 10\%$ for a chemical method. Glucose, fructose, maltose, mannitol, mannose, sucrose and trehalose interfered, but nine other carbohydrates tested did not. The method is applicable to culture fluids after bacterial growth. M. A. Ellis

161. **Microbiological assay of glucose as applied to starch hydrolysates.** M. D. Smith, M. W. Radomski and J. J. Kagan (Univ. Toronto, Ontario, Canada). *Anal. Chem.*, 1960, **32** (6), 678–680.—Th

thod is based on the ability of *Lactobacillus casei* ferment glucose selectively in a mixture of the gars found in starch hydrolysates. The response determined by titration with standard alkali in. and the dose - response curve is linear up to ng of glucose. A heavy-inoculum technique ortens the incubation time to 2 hr. and is sensitive small quantities of glucose. Recoveries of ucose added to hydrolysates averaged 100·1% at e 1-mg level, and a mean difference of ±3·0% th a standard deviation of 0·148 was obtained om 50 sets of duplicate titrations. G. P. Cook

162. **Gas-chromatographic retention time of formdehyde.** A. I. Schepartz and P. E. McDowell gric. Res. Service, U.S. Dept. of Agric., Philalphia, Pa.). *Anal. Chem.*, 1960, **32** (6), 723.—A tention time of 2·3 min. was obtained for formldehyde on a 10-ft. column of Carbowax 20M on romosorb (35 to 80 mesh) at 90°, with a helium w-rate of 32 ml per min. In determining this cention time the flash-exchange technique of Ralls *nal. Abstr.*, 1960, **7**, 4825) was used.
K. A. Proctor

163. **Separation and identification of C8 aldehydes. se of gas - liquid chromatography, nuclear magnetic sonance, and infra-red spectroscopy.** J. S. Matews, F. H. Burow and R. E. Snyder (Gulf Res. d Devel. Co., Pittsburgh, Pa., U.S.A.). *Anal. em.*, 1960, **32** (6), 691–693.—In the method scribed, individual isomers or groups of isomers e separated by preparative gas chromatography. uclear magnetic resonance is then used to deterne the number of CH_3, CH_2 and CH groups, and .. spectroscopy to indicate the presence of $(CH_2)_n$ oups ($n \not< 3$). The aldehyde is finally reduced to hydrocarbon, which is identified by comparison th suitable standards by gas - liquid chromatoaphy. K. A. Proctor

164. **Chromatographic separation of 2,4-dinitroenylhydrazine derivatives of highly oxygenated rbonyl compounds.** M. L. Wolfrom and G. P. rsenault (Ohio State Univ., Columbus, U.S.A.). *nal. Chem.*, 1960, **32** (6), 693–695.—The chromatoaphic separation of the 2,4-dinitrophenylhydrazine rivatives of highly oxygenated 2- and 3-carbon rbonyl compounds was effected on a column of icic acid - Celite (5:1, containing 8% of H_2O).
G. P. Cook

165. **Paper chromatography and structural relaonships of organic acids.** J. R. Howe (Low Temp. es. Sta., Cambridge, England). *J. Chromatoaphy*, 1960, **3** (5), 389–405 (in English).—The and R_M [$= \log(1/R_F - 1)$] values for 111 organic ids were determined in an alkaline and an acid lvent, *viz*, n-propanol - 2 N aq. NH_3 (7:3) and propanol - aq. satd. soln. of SO_2 (7:3). Results e discussed in relation to the structures of the ids, which included several homologous series. plot of R_F values in acid solvent against those in kaline solvent shows that distinct groupings are tained according to the number of carboxyl oups in the molecule, whereas neutral compounds ve similar R_F values in both solvents. The M values for several homologous series when otted against number of carbon atoms give pprox. linear relationships up to 8 carbon atoms in th solvents. Definite groupings also occur if the fference between the R_M value in alkaline solvent d that in acid solvent is plotted against the R_M lues in alkaline solvent, and homologous series rm sub-groups. A study of the R_M value could therefore be used to give an indication of the presence of substituent groups such as $-CO_2H$, –OH, alkyl, $-NH_2$ and bromo– in an unknown acid and the manner in which they are arranged.
S. M. Marsh

166. **Gas-chromatographic separation of volatile organic acids in presence of water.** I. R. Hunter, V. H. Ortegren and J. W. Pence (U.S. Dept. Agric., Albany, Calif., U.S.A.). *Anal. Chem.*, 1960, **32** (6), 682–684.—Volatile organic acids (C_2 to C_8) are separated at 125° by using an adipate polyester of diethylene glycol (LAC-1-R 296) as the liquid phase and He as the carrier gas. The fractions emerging from the column are oxidised to CO_2 which is then measured in a thermal conductivity cell; H_2O does not affect the separation, and is removed by a drying tower placed before the conductivity cell.
G. P. Cook

167. **Determination of oxalates in the presence of chromium.** V. F. Saksin (Technol. Inst. Yaroslav). *Zavod. Lab.*, 1960, **26** (5), 546–547.—An accurately measured vol. of 0·1 N $KMnO_4$ is treated with excess of $MnSO_4$ followed, after 5 min., by 15 to 20 ml of 2 N H_2SO_4 to give H_2MnO_3, which will oxidise oxalate but not Cr^{3+}. The soln. is boiled for 20 to 25 min. with a known vol. of the soln. to be analysed, then cooled, mixed with sufficient 20% KI soln. to react with the excess of H_2MnO_3, and titrated with 0·1 N $Na_2S_2O_3$. G. S. Smith

168. **Differential thermal analysis and thermogravimetry of some salts of guanidine and related compounds.** M. I. Fauth (U.S. Naval Propellent Plant, Indian Head, Md.). *Anal. Chem.*, 1960, **32** (6), 655–657.—The picrates and styphnates of hydrazine, guanidine, aminoguanidine, guanylurea, *N*-methylguanidine and *N*-ethylguanidine were prepared and their impact sensitivities and thermal behaviour under rapid heating studied. The temp. range covered was from room temp. to the point of decomposition of the compound being investigated. Differential thermal analysis curves and thermogravimetric results are given for 16 compounds. At rapid heating rates (8° per min.) considerably lower decomposition temp. may be expected than would be indicated by m.p. and other values obtained by slow heating. G. P. Cook

169. **Colour reactions for organophosphorus compounds.** H. Hecker and F. Hein (Inst. f. Anorg. Chem., Friedrich Schiller Univ., Jena). *Z. anal. Chem.*, 1960, **174** (5), 354–359 (in German).—(*a*)—Phosphine oxides and dialkylphosphinic acids give deep-blue colorations when heated with anhydrous $NiBr_2$ in toluene. Some phosphine sulphides and tertiary phosphines give green colours. Acetone and ethyl methyl ketone interfere. (*b*)—Alkylphosphine oxides and tetra-alkylphosphonium salts give a deep-violet colour with *m*-dinitrobenzene and 35% NaOH soln. (*c*)—Tertiary phosphines give a red - violet colour with 3,5-dinitrobenzoic acid, triethylphosphine oxide and CCl_4. (*d*)—Aliphatic phosphine sulphides give a green colour changing to blue when heated on a water bath with vanillin and conc. HCl. (*e*)—Aliphatic phosphine oxides give a brown or green coloration with CS_2, NaOH and a molybdate. W. T. Carter

170. **Detection of phosphate esters on paper chromatograms.** F. E. G. Harrap (Levington Res. Sta., Ipswich, Suffolk, England). *Analyst*, 1960, **85**, 452.—Phosphate esters, *e.g.*, fructose 1,6-diphosphate, glucose 1-phosphate, glucose 6-phos-

phate and phytic acid are usually detected on filter-paper by hydrolysis to H_3PO_4, which is treated with an acid molybdate spray or dip; the complex formed is then reduced to a blue compound when the paper is treated with H_2S or exposed to u.v. light. After either treatment the paper gradually becomes blue, and later identification of faintly coloured spots becomes difficult. Also certain naturally occurring acids (*e.g.*, citric, tartaric and ascorbic acids) form a blue colour slowly. To overcome these disadvantages the filter-paper is dried in air and dipped in a mixture of 5 ml each of 60% $HClO_4$ and 20% molybdate soln., 10 ml of N HCl and 80 ml of acetone. The paper, dried in air, is exposed to u.v. radiation then immediately dipped in a 2·5% (w/v) soln. of benzoin α-oxime in methanol and dried. This provides the paper with a background of a white ppt. stable for some weeks. The acids (*supra*) in 1% soln. do not form visible spots on this background. A. O. Jones

171. **Rapid method for qualitative analysis of volatile mercaptan [thiol] mixtures.** J. F. Carson, W. J. Weston and J. W. Ralls (Western Utilisation Res. & Devel. Div., U.S. Dept. Agric., Albany, Calif.). *Nature*, 1960, **186**, 801.—Equal weights (3 to 5 mg) of a mixture of mercuric mercaptides and warm toluene-3,4-dithiol are centrifuged to produce a slurry in a capillary tube. The tube is connected to a gas-chromatography unit and the lower end of the tube is heated at 245° to 260° for 15 sec. when an exchange reaction occurs and C_1 to C_4 saturated thiols flash into the unit for separation and identification. Only highly volatile thiols give good detector response. A. C. R. Hartley

172. **A new volumetric method for the estimation of thiourea.** Prakash Chandra Gupta (Coll. of Sci., Hindu Univ., Varanasi). *J. Indian Chem. Soc.*, 1960, **37** (4), 213–216.—Thiourea is quant. oxidised to urea and sulphate by iodine in the presence of $NaHCO_3$. Standard iodine soln. of suitable concn. (20 ml) is mixed with 10% $NaHCO_3$ soln. (20 ml) and titrated with thiourea soln. The end-point may be detected visually (starch indicator) or potentiometrically. Alternatively, the thiourea soln. may be mixed with $NaHCO_3$ soln. and a known excess of iodine soln. and the excess back-titrated potentiometrically with standard arsenous oxide soln. R. E. E.

173. **Analysis of dithiocarbamates.** G. Balif (Inst. Agron. Res., Acad. R.P.R.). *Rev. Chim., Bucharest*, 1960, **11** (6), 338–341.—A review of current methods is given, with 18 references. H. Sher

174. **Determination of water-extractable sodium in liquid hydrocarbons.** K. H. Nelson and M. D. Grimes (Phillips Petroleum Co., Bartlesville, Okla., U.S.A.). *Anal. Chem.*, 1960, **32** (6), 594–595.—The Na is extracted by shaking the hydrocarbon with an equal volume of water and is determined in the aq. phase by flame photometry. The relative error is ±4% and the precision is ±2%, when applied to samples boiling in the range 135° to 900° F. G. P. Cook

175. **Colorimetric determination of benzene and chlorobenzene when present together.** M. Mirjolet and C. Domange (Univ. Nancy, France). *Bull. Soc. Pharm. Nancy*, 1959, (41), 23–29.—Mixtures of benzene and chlorobenzene (**I**) are sometimes used as solvents for gum rubber in rubber cements. Nitration yields *m*-dinitro-derivatives in both cases, and when the product is dissolved in ethyl methyl ketone and shaken with conc. aq. KOH soln. a violet colour appears. The max. absorption produced by benzene is at 570 mμ, while the max. of the weaker absorption produced by **I** is at 540 mμ. Attempts to distinguish colorimetrically between benzene and **I** have not given satisfactory results. However, it was found that, by boiling with Na_2CO_3, the 1-chloro-2,4-dinitrobenzene produced by nitration of **I** was completely converted into 2,4-dinitrophenol, which gave max. absorption at 420 mμ, permitting sharp differentiation from benzene, since 2,4-dinitrobenzene was not affected by the Na_2CO_3. For small amounts of benzene and **I** (singly or together) the extinctions of the coloured soln. followed the Beer - Lambert law. Chem. Abstr.

176. **Determination of polycyclic aromatic hydrocarbons in tobacco smoke.** A. Candeli, A. J. Lindsey and K. Persaud (Dept. of Chem., Sir John Cass College, London). *Anal. Chim. Acta*, 1960, **22** (5), 458–461.—Previously described apparatus and methods (chromatographic and u.v. spectrophotometric) (*cf.* Lindsey, *Anal. Chim. Acta*, 1959, **20**, 175; **21**, 101; and Commins *et al.*, *Brit. J. Cancer*, 1955, **9**, 304) have been improved. R. M. S. Hall

177. **Relative detector response in gas chromatography. II. Benzene hydrocarbons, phenols and phenol ethers.** G. R. Jamieson (Chem. Dept., Paisley Tech. Coll., Scotland). *J. Chromatography*, 1960, **3** (5), 494–496 (in English).—Determination of the relative detector response of homologous series of benzene hydrocarbons, phenols and phenol ethers shows that there is an increase in response with an increase in molecular weight and for isomers there is a decrease in response with increase in chain-branching. Lower relative detector responses are given by *ortho*-isomers than by either *meta*- or *para*-isomers. If a compound has a group that forms part of a side-chain its response is similar to that of the isomer that has the group attached to the benzene ring. The introduction of a hydroxyl or carboxyl group lowers the relative detector response, but the introduction of a chloro-group into a phenol or phenol ether has little effect. S. M. Marsh

178. **Neutral silver nitrate as a reagent in the chromatographic characterisation of phenolic compounds.** W. J. Burke, A. D. Potter and R. M. Parkhurst (Univ. Utah, Salt Lake City, U.S.A.). *Anal. Chem.*, 1960, **32** (6), 727–728.—A neutral soln. of $AgNO_3$ in aq. acetone gives characteristic colour reactions with various phenols and certain of their ether derivatives. The time required to develop the colour is also a characteristic of the compound involved. R_F values in 5 solvent systems, colours produced, and colour-development times for 37 substances are listed. G. P. Cook

179. **Infra-red spectra of 1,4-benzoquinones.** R. L. Edwards, J. Keighley and D. G. Lewis (Inst. Technol., Bradford, England). *J. Appl. Chem.*, 1960, **10** (6), 246–249.—Data are given for twenty 1,4-benzoquinones. Some derivatives of these compounds occur as pigments in fungi.

180. **Simple specific test for inner-ring o-quinones.** E. Sawicki and W. Elbert (Air Pollution Engng Res., Robert A. Taft San. Engng Centre, Cincinnati, Ohio, U.S.A.). *Anal. Chim. Acta*, 1960, **22** (5), 448–451.—The quinone is treated with 3,4-dimethoxyaniline in hot acetic acid to give blue to green

mpounds absorbing at 590 to 615 mμ. A spot-t procedure is described. Benzil, chloranil, orenone, acenaphthenequinone, anthraquinone l benzanthrone give negative results; 1,4-phthaquinones give a scarlet coloration. The ntification limits are 5 to 20 μg. The results of our tests with *o*-quinones in H_2SO_4 are also orted. R. M. S. Hall

181. **Determination of aniline in *N*-ethylaniline.** B. Whetsel, W. E. Roberson and M. W. Krell nnessee Eastman Co., Kingsport, U.S.A.). al. Chem., 1960, **32** (6), 730.—Infra-red data are en (*cf. Anal. Abstr.*, 1959, **6**, 1380).

182. **Analysis of mixtures of aniline and *N*-ethyl-line.** K. B. Whetsel, W. E. Roberson and M. W. ell (Tennessee Eastman Co., Kingsport, U.S.A.). al. Chem., 1960, **32** (6), 730.—Infra-red data are en (*cf. Anal. Abstr.*, 1959, **6**, 1380).

183. **Analysis of mixtures of *N*-ethyl-*m*-toluidine l *N*-(2,3-dihydroxypropyl)-*N*-ethyl-*m*-toluidine.** B. Whetsel, W. E. Roberson and M. W. Krell nnessee Eastman Co., Kingsport, U.S.A.). al. Chem., 1960, **32** (6), 730.—Infra-red data are en (*cf. Anal. Abstr.*, 1957, **4**, 4028).

184. **Photometric titration of weak bases in non-ueous media.** L. E. I. Hummelstedt and D. N. me (M.I.T., Cambridge, Mass., U.S.A.). *Anal. em.*, 1960, **32** (6), 576–581.—Weak organic bases determined by photometric titration with lO_4 in acetic acid as the titrant and acetic acid or thyl cyanide as the solvent. Differential titra-n of 4 components in a mixture is possible. ration is normally carried out at the 10^{-2} M el but many bases can be successfully measured the 10^{-4} M level. Wavelength data are given 18 aromatic compounds. The deviation is erally $< \pm 5\%$. G. P. Cook

185. **Studies on the analysis of drugs by the use of a-red spectra. II. Assay of naphthols.** Michio uki, Eizo Nakamura and Yuzo Nagase (Coll. of arm., Kashiwagi, Shinjuku-ku, Tokyo). *J. arm. Soc. Japan*, 1959, **79** (9), 1145–1148.—The ermination of 1- (**I**) and 2-naphthol (**II**) in a ary mixture was attempted by infra-red spectra. determination was made in CS_2 and in KBr s by means of the bands at 770, 790 and 1083 $^{-1}$ for **I** in CS_2, 765 and 790 for **I** in KBr, 744 and for **II** in CS_2, and 815 and 843 for **II** in KBr. determination of **I** in **II** was unsuccessful in , owing to the slight solubility of **II**. The orption of **II** in the soln. at 837 mμ is variable ng to its intermolecular action with **I**. The ndard deviations were $<1{\cdot}18\%$ and no significant erence was found between results obtained with soln. and KBr discs. S. Natori

186. **Fluorimetry. III. Fluorimetric determina-of naphthionic acid by means of short-wave a-violet light (313 mμ).** J. Eisenbrand and H. ver (Chem. Untersuchungsamt, Saarbrücken, rland). *Z. anal. Chem.*, 1960, **174** (6), 414–418.— absorption spectrum of naphthionic acid aphthylamine-4-sulphonic acid] (**I**) has a max. ween 315 and 325 mμ and the fluorescence trum has a max. at 425 mμ. The Hg line at mμ is useful for fluorescence excitation. Full rescence is attained between pH 6 and 8, and log fluorescence *vs.* log concn. curve is linear in concn. range $1{\cdot}6 \times 10^{-9}$ to 1×10^{-6} g per ml. The method is used for determining **I** in the presence of 2-amino-1-naphthol-4-sulphonic acid in the enzymic degradation of Azorubine S (C.I. Food Red 3). The sensitivity is 0·001 μg per ml. J. P. Stern

187. **The separation of some terpenoid compounds by gas - liquid chromatography.** E. von Rudloff. *Canad. J. Chem.*, 1960, **38** (5), 631–640.—The gas - liquid chromatographic separation of mixtures of some terpene hydrocarbons, their oxygenated derivatives, two sesquiterpene alcohols and three monophenols on a variety of columns was studied. The best solid support was Chromosorb W, a flux-calcined diatomaceous earth, which enabled separation to be made at high temp. without causing decomposition of the compounds. The effect of temp. and sample size on the degree of separation was considerable; low temp. gave the best results and sample sizes above 3 μl caused delays in the retention times and broadened the peaks. Monoterpene hydrocarbons were best separated on columns with squalene, Carbowax 400, Craig polyesters, and polyphenyl ether as the liquid phases at temp. below 130°. At higher temp. the polyester and polyphenyl ether columns were most suitable for the separation of oxygenated terpenes, sesquiterpene alcohols and monophenols. The adipic acid - polyoxyethylene glycol polyester column was the most suitable for all the compounds tested. G. P. Cook

188. **Modified Ehrlich's reagent for measurement of indolic compounds.** M. Knowlton, F. C. Dohan and H. Sprince (Veterans Admin. Hosp., Coatesville, Pa., U.S.A.). *Anal. Chem.*, 1960, **32** (6), 666–668.—The sensitivity and stability of the Ehrlich reaction are improved by the use of a nearly saturated soln. of *p*-dimethylaminobenzaldehyde in 12 N HCl as the reagent. Absorbance data are given for several indolic compounds and the concn. curves follow Beer's law, except that for indole, which deviates slightly. Concn. as low as $2{\cdot}5 \times 10^{-6}$ mole can be determined and the coeff. of variation range from 0·29 to 5·68. G. P. Cook

189. **Determination of phenothiazine in commercial preparations by chromatography.** D. Gunew (Central Res. Lab., Imperial Chemical Industries of Australia and New Zealand Ltd., Newson St., Ascot Vale, Melbourne, Australia). *Analyst*, 1960, **85**, 360–364.—Sufficient sample to produce 25 to 35 mg of phenothiazine per ml of final soln. is extracted in a modified Soxhlet apparatus (described) with dichloromethane. The column is a mixture of silica gel (15 g), Magnesol (15 g), copper powder (1 g, prep. described) and the solvent mixture [dichloromethane - n-hexane (1 : 9)] (120 ml) in a water-jacketed chromatographic tube maintained at 14° to 16° surmounted by a reservoir of the solvent mixture. The sample soln. (2 ml) is applied to the column by means of a delivery tube, and the column is eluted under pressure of nitrogen with 800 ml of the solvent, and 10-ml fractions are collected. The solvent is evaporated from each fraction individually under reduced pressure at 30° to 35°. The phenothiazine-containing fractions (recognisable visually) are rinsed with dichloromethane into a tared flask, the solvent is removed and the residue is weighed and its m.p. determined. Since Magnesol is not now obtainable, Florisil (200 to 300 mesh) can be used in its place. A. O. Jones

190. **New photometric method for the determination of diphenylamine in phenothiazine.** V. Pelloni and M. Sterescu. *Rev. Chim., Bucharest*, 1960, **11** (6), 346.—The proposed method is based on the colour reaction of diphenylamine (**I**) with *p*-dimethylaminobenzaldehyde (**II**) in the presence of oxidising agents. *Procedure*—A standard curve is prepared by dissolving 0·09 g of pure phenothiazine and 0·01 g of **I** in 100 ml of methanol - water (1:4). The soln. is centrifuged and filtered, and aliquots corresponding to 25 to 100 μg of **I** are taken. To each is added 2 ml of 10 N H_2SO_4, 4 ml of reagent (38 ml of conc. H_2SO_4, 37 ml of water and 0·125 g of **II**) and 6 drops of H_2O_2 soln. (5 ml of water and 2 drops of H_2O_2). The extinction is read after 20 min. at 570 mμ in a 1-cm cell against water. For the determination, 0·1 g of crude phenothiazine is dissolved in 100 ml of aq. methanol and treated as described above, and the extinction is compared with the standard curve. The error is $\pm$5%.
H. Sher

191. **Determination of 1,2-diphenylpyrazolidine-3,5-dione and its derivatives.** M. Konupčík, F. Kupčík and M. Liška (Dept. Tech. Control, Farmakon, Olomouc, Czechoslovakia). *Československ. Farm.*, 1960, **9** (5), 236–238.—Two acidimetric methods and one bromimetric method are described for the titration of 1,2-diphenylpyrazolidine-3,5-dione and 4-(3-oxobutyl)-1,2-diphenylpyrazolidine-3,5-dione (**I**) in non-aqueous solvents. *Acidimetric procedures*—(*i*) The sample (0·2 to 0·3 g) is dissolved in $CHCl_3$ or dioxan (20 ml, neutralised to phenolphthalein) and titrated with 0·05 N Na methoxide, with phenolphthalein as indicator. (*ii*) The sample (0·4 to 0·5 g) is dissolved in dioxan (25 ml) and titrated with 0·1 N NaOH. The errors are $\pm$0·15% and 0·05%, respectively. *Bromimetric procedure*—The sample (0·2 to 0·4 g) is dissolved in glacial acetic acid, conc. HCl (5 ml) and 20% KBr soln. (5 ml) are added, and the mixture is titrated potentiometrically with 0·1 N $KBrO_3$ (*cf.* Jančík *et al.*, *Anal. Abstr.*, 1958, **5**, 246). The error is 0·1%. Only the bromimetric procedure is satisfactory for the determination of the sodium salt of **I**.
J. Zýka

192. **Separation of 1,2-diphenylpyrazolidine-3,5-dione derivatives by paper chromatography.** M. Konupčík, F. Kupčík and M. Liška (Dept. Tech. Control, Farmakon, Olomouc, Czechoslovakia). *Československ. Farm.*, 1960, **9** (5), 239–241.—*Procedure*—Place the soln. of the sample (50 to 100 μg) in $CHCl_3$ on Whatman No. 1 paper and then impregnate the paper with formamide - ethanol (1:1) containing 5% of ammonium formate. After 10 min. separate chromatographically with benzene - cyclohexane (4:1 or 3:2); after 3 hr. dry *in vacuo* at 50° and detect by spraying with a freshly prepared mixture of 15% $FeCl_3$ soln. and 1% $K_3Fe(CN)_6$ soln. (1:1). R_F values are tabulated.
J. Zýka

193. **New method for the determination of pyrazolanthrone (6-oxoanthra[1,9-*cd*]pyrazole).** M. Matrka and F. Navrátil (Res. Inst. Org. Synth., Pardubice-Rybitví, Czechoslovakia). *Chem. Průmysl*, 1960, **10** (5), 248–249.—*Procedure*—Dissolve 6-oxoanthra-[1,9-*cd*]pyrazole (0·05 g) in ethanol (10 ml) and N NaOH (10 ml) and dilute with H_2O to 100 ml. Dilute 1 ml of this soln. to 10 ml with Britton - Robinson buffer soln. (pH 11·98), remove O with a stream of N and register the polarographic wave ($E_{\frac{1}{2}} = -1{\cdot}12$ V). Compare with a calibration curve.
J. Zýka

194. **Molecular sieve adsorption. Application to hydrocarbon-type analysis.** J. G. O'Connor and M. S. Norris (Gulf Res. and Dev. Co., Pittsburgh, Pa., U.S.A.). *Anal. Chem.*, 1960, **32** (6), 701–706.—Normal hydrocarbons are determined in petroleum distillates boiling between 100° and 650° F by means of powdered molecular sieves, type 5-A, without previous fractionation of the distillates into narrow boiling-ranges. The sample is weighed into a column containing the sieves and the non-adsorbed hydrocarbons are eluted with isopentane. The adsorbed hydrocarbons are determined by weighing the column after removal of the excess of eluent, and can be removed from the column by extraction into n-pentane. Various fractions are analysed by mass spectrometry. A schematic diagram illustrates the various steps in the analysis of samples in conjunction with a silica gel separation technique. The accuracy, as standard deviation, is within $\pm$0·8% of the normal hydrocarbon content of the sample.
G. P. Cook

195. **Polarographic determination of manganese in gasoline. Triethanolamine complexes of manganese(II and III) and lead(II).** E. R. Nightingale, jun., G. W. Wilcox and A. D. Zielinski (Univ. of Nebraska, Lincoln, U.S.A.). *Anal. Chem.*, 1960, **32** (6), 625–628.—The organomanganese compounds are decomposed by exposing the sample to u.v. light while it is being heated under reflux with conc. HCl. The Mn in the aq. extract is then determined polarographically as the triethanolamine complex at $-0{\cdot}7$ V *vs.* the S.C.E.; Pb is determined at $-0{\cdot}6$ V *vs.* the S.C.E. on another portion of the extract. When applied to petroleum-base stocks the standard deviation from the amount present was 5·2% for Mn and 1·6% for Pb. For pure iso-octane or aq. standards the average deviation was $< \pm$0·5%.
G. P. Cook

196. **Determination of polynuclear aromatic hydrocarbons in petroleum waxes.** L. B. Nelson and D. J. Stormont (Socony Mobil Oil Co., Brooklyn, N.Y., U.S.A.). *Chem. & Ind.*, 1960, (20), 561.—A procedure is given for the separation and determination of tetracyclic and higher aromatic hydrocarbons in concn. of 10^{-8} to 10^{-9} in petroleum wax, with recoveries of 45 to 65%. The molten wax (100 g) is passed through a heated column (15 cm × 4·5 cm) of activated Al_2O_3 or silica gel which retains the polycyclic aromatics. After elution of the residual wax with iso-octane and extraction (in a Soxhlet thimble) of the extruded adsorbent with benzene - ethanol under N, the polycyclic aromatics can be further concentrated by making five partition extractions of the residue (dissolved in 50 to 100 ml of cyclohexane) in the thimble with nitromethane. The residue from the partition is then submitted to one-dimensional ascending paper chromatography with methanol - ethyl ether - water (4:4:1) as solvent on S. & S. 2043*b* acetylated paper. Sections of the chromatogram are cut out, eluted with cyclohexane - ethanol (1:1) and the u.v. spectra of the extracts are recorded. When necessary, fractions are re-examined by chromatography on Whatman No. 1 paper, with an iso-octane - dimethylformamide system, followed by comparison of R_F values (referred to benzo[*a*]pyrene), fluorescence colour and u.v. spectra.
W. J. Baker

197. **Separation of polycyclic aromatic hydrocarbons in complex mixtures. Chromatographic determination of trace amounts in petroleum waxes.** W. Lijinsky (Chicago Med. Sch., Ill., U.S.A.).

al. Chem., 1960, **32** (6), 684–687.—Four polyclic aromatic hydrocarbons, *viz*, benzo[*a*]pyrene, benz[*a,h*]anthracene, benz[*a*]anthracene and chrysene, added to petroleum waxes both individually and combined, were recovered by chromatography on magnesium oxide mixed with Celite, followed by paper chromatography with different solvent systems. Recovery ranged from 35% for benzo[a]pyrene to 94% for dibenz[*a,h*]anthracene in the concn. range 0·01 to 1 p.p.m. G. P. Cook

198. **Direct determination of traces of total oxygen in naphthas.** I. J. Oita (Res. and Develop. Dept., Standard Oil Co., Whiting, Ind., U.S.A.). *Anal. Chim. Acta*, 1960, **22** (5), 439–443.—Further amendments to the modified Schütze method (*cf.* Oita and Conway, *Anal. Chem.*, 1954, **26**, 600) have been made. These include the handling of the sample in a quartz hollow spiral, controlled magnetically. Full details of the modified pyrolysis tube and operating instructions are given. For a synthetic mixture (cetyl alcohol in iso-octane) the value found was 157 ± 14 p.p.m. (3 determinations), calculated 144. For routine samples the deviations from the mean (duplicates) were about ±20%.
R. M. S. Hall

199. **Identification of Teepol and sodium lauryl sulphate in a commercial detergent mixture.** B. Sewell. *Lab. Practice*, 1960, **9** (6), 381.—About 1 g of mixed alcohols, obtained by hydrolysing the detergent mixture by heating it under reflux with N HCl for 30 min., is dissolved in 15 ml of dry benzene and heated under reflux for 2 hr. with 0·5 g of 3,5-dinitrobenzoyl chloride and 5 ml of pyridine. The mixture is dissolved in ethyl ether (50 ml) and extracted several times with 6 N HCl to remove pyridine and then with 4 N NaOH to remove 3,5-dinitrobenzoic acid. The ether soln. is washed free from alkali with water, dried over Na_2SO_4, and evaporated to dryness. The ester is then crystallised from light petroleum - ethanol (1 : 4). About 0·05 g of the crystals is dissolved in 1 ml of benzene - acetone (1 : 1) and paper chromatography is carried out with benzene as solvent until the front has ascended by 15 to 20 cm. After drying, the paper is sprayed with satd. ethanolic hydroxyammonium chloride soln. and dried. A mixed conc. ethanolic NaOH soln. is then sprayed on. A yellow band near the solvent front indicates sodium lauryl sulphate in the original sample; a sharp yellow band near the base-line indicates Teepol in the original product. The presence of an alkylarylsulphonate does not cause interference, since it is not hydrolysed under these conditions. N. E.

200. **Methods of detection of the adulteration of volatile oils with ethanol.** B. Kamiński and O. Motkowska (Drug Res. Inst., Warsaw). *Acta Pol. Pharm.*, 1960, **17** (3), 213–219.—The methods in Pharm. Polon. III for the detection of ethanol in volatile oils are critically studied. A review of other published methods is given. The best test for the detection of a small amount of alcohol in essential oils is that of Pharm. Ned. VI, *viz*, the determination of refractive indices before and after removal of ethanol from the sample by shaking with satd. aq. NaCl soln. and drying with anhyd. Na_2SO_4. If the values differ by >0·001 the presence of ethanol is indicated. (19 references.)
B. K.

201. **Analysis and composition of oil of lemon by gas - liquid chromatography.** R. A. Bernhard (Dept. of Food Sci. and Technol., Univ. of Calif., Davis, U.S.A.). *J. Chromatography*, 1960, **3** (5), 471–476 (in English).—Cold-pressed Californian lemon oil has been analysed by gas - liquid chromatography with LAC-2-R446 (adipate polyester of diethylene glycol partially cross-linked with pentaerythritol) and LAC-4-R777 (succinate polyester of diethylene glycol) as stationary liquid phases. Compounds identified by the corrected retention volumes method were—α-pinene, β-pinene, myrcene, (+)-limonene, n-heptanal, 1,8-cineole, γ-terpinene, n-hexanol, n-octanol, methylheptenone, n-nonanal, linalol, n-decanal, citronellyl acetate, citronellal, n-undecanal, tetrahydrogeraniol, decyl acetate, geraniol, α-terpineol, citronellol, neral, geranyl acetate, citral and (+)-carvone.
S. M. Marsh

202. **Determination of carbonyl groups in cellulose.** H. Stübchen-Kirchner (Inst. f. Chemie u. chem. Technol. des Papieres u. des Zellstoffes, Tech. Hochschule, Graz). *Öst. ChemZtg*, 1960, **61** (5), 132–141.—A comprehensive review, with 60 references, is presented. S. M. Marsh

203. **Rapid method for the determination of the acetic acid content of cellulose acetates.** K. H. Bischoff and K. J. Linow (Dtsch. Akad. Wiss., Berlin). *Faserforsch. u. Textiltech.*, 1960, **11** (5), 245.—In Batt and Penche's method (*Bull. Soc. Chim. France*, 1929, **45**, 152) the cellulose ester is dissolved in pyridine, but cellulose acetates with high contents (59 to 62·5%) of acetate are not completely soluble. This can be overcome by shaking the cellulose acetate (0·4 g) with dichloromethane (20 ml) for 30 min. then adding pyridine (40 ml) and 0·5 N NaOH (30 ml). N. E.

204. **Analysis of fibre mixtures.** T. Szentpály, R. Szentpály and A. Stark (Res. Inst. of the Textile Ind., Budapest). *Faserforsch. u. Textiltech.*, 1960, **11** (4), 189–195.—Methods are described for the quant. analysis of mixtures containing two or more of the following types of fibre, *viz*, wool, regenerated protein, cotton, cellulose rayon, polyamide, polyester and poly(vinyl cyanide), by determining the losses in weight caused by treating the sample with a selective solvent for a single type of fibre, or with a number of different solvents in succession. Full details are given of these solvents, and tables show their applicability to all possible 2 to 4-component mixtures of these types of fibres.
H. L. Whitehead

205. **Reflectance spectrophotometric determination of soluble iron in delustered acrylic fibres.** M. E. Gibson, jun., D. A. Hoes, J. T. Chesnutt and R. H. Heidner (Chemstrand Corp., Decatur, Ala., U.S.A.). *Anal. Chem.*, 1960, **32** (6), 639–642.—The sample is dissolved in dimethylformamide, the Fe is reduced with hydroxyammonium chloride, and a coloured complex is prepared with a soln. of 1,10-phenanthroline in dimethylformamide. The presence of TiO_2 in the sample renders the soln. turbid or translucent, so that transmittance measurements are inapplicable, and the colour is measured by reflectance at 510 and 610 mμ, with a magnesium oxide reference standard, and corrected for a blank and for the Fe in the reagents. The difference between the corrected readings at 510 and 610 mμ is referred to a calibration curve. The standard deviation (8 determinations) for 2 p.p.m. of Fe was ±0·20. G. P. Cook

206. **Determination of the fluorine content of high polymers.** E. Schröder and U. Waurick (Inst. f. Chem. Tech. der Plaste, Leipzig). *Plaste u. Kautsch.*, 1960, **7** (1), 9–11.—The polymer is heated with sodium in a nickel crucible, the excess of sodium is destroyed, and the aq. solution of the product is passed through an ion-exchange column. The F content of the percolate is determined by titration with 0·1 N alkali. Any Cl present may be determined argentimetrically. L. A. O'Neill

207. **Determination of monomer in polystyrene and finished products.** J. Eisenbrand and H. W. Eich (Chem. Untersuchungsamt f.d. Saarland, Saarbrücken). *Z. anal. Chem.*, 1960, **175** (1), 4–9 (in German).—The sample (0·1 g) is dissolved in 50 ml of $CHCl_3$, and 10 ml of the soln. is added slowly from a burette to a flask containing 70 to 80 ml of methanol. The soln. is shaken, diluted to 100 ml with methanol and again shaken, then set aside for 16 hr. The dissolved monomer is filtered off from the pptd. polymer and the extinction of the clear soln. is measured at 245 mμ. The calibration curve, prepared from known soln. of freshly distilled styrene dissolved in methanol, is linear. The max. error is 0·03%. B. B. Bauminger

208. **Analysis of simple alkyd resins.** E. Schröder and K. Thinius (Inst. für Chem. Tech. der Plaste, Leipzig). *Dtsch. Farben-Z.*, 1960, **14** (4), 144–148; (5), 189–193.—Fatty acids recovered from alkyd resins have been examined by reversed-phase chromatography on paper impregnated with liquid paraffin, and acetic acid (90%, saturated with paraffin oil) as the mobile phase. Alkyds prepared from drying and non-drying oils were examined, but in several cases the spots obtained from the recovered fatty acids were different from those of the acids in the parent oil, and the method could not be considered as generally reliable for identifying the oil component of an alkyd. Procedures for the determination of dibasic acids, fatty acids and unsaponifiable matter, which are carried out after saponification with Na ethoxide, are given. The most convenient method for the determination of polyhydric alcohol involves aminolysis with ethanolamine and separation and oxidation of the alcohol. L. A. O'Neill

209. **Identification of the glycol components of unsaturated polyester resins by paper chromatography.** H. Rejhová and W. Ulbrich (Forschungsinst. f. Kunstharze, Pardubice, Czechoslovakia). *Plaste u. Kautsch.*, 1959, **6** (11), 539–540.—The resin is saponified with ethanolic KOH, the K salts of the dibasic acids are filtered off, the alkali is neutralised with CO_2, the K_2CO_3 and the ethanol are removed,and the glycols are examined by ascending paper chromatography, with n-butanol saturated with H_2O as the mobile phase; the spots are revealed with ammoniacal $AgNO_3$. Results obtained on polyesters agreed with those obtained by conversion of the glycol into a 3,5-dinitrobenzoate or diphenylcarbamate derivative for identification. L. A. O'Neill

210. **Determination of water in pigment pastes and printing ink colours.** F. Öhme (Inst. zur Entwicklung Analysenmethoden, Weilheim/Obb, Germany). *Farbe u. Lack*, 1960, **66** (3), 142–145.—The material is extracted with dioxan; the dielectric constant of the dioxan extract is determined before and after passage through a column of a drying agent, and the difference is a measure of the H_2O present. L. A. O'Neill

211. **Volumetric analysis with complexans in the paint laboratory.** O. Borchert. *Plaste u Kautsch.*, 1959, **6** (11), 562–568.—Applications of complexometric methods to the analysis of paint compositions, *e.g.*, the determination of metals present in pigments and in driers, are reviewed. L. A. O'Neill

212. **Determination of chlorendic acid in fire-retardant paint.** G. G. Esposito and M. H. Swann (Aberdeen Proving Ground, Md., U.S.A.). *Anal. Chem.*, 1960, **32** (6), 680–681.—The sample is dissolved in benzene and saponified with KOH in isopropyl alcohol. The resulting dipotassium salt of chlorendic acid (3,6-endodichloromethylene-3,4,5,6-tetrachloro-Δ^4-tetrahydrophthalic acid) (**I**) is filtered off and dissolved in H_2O; the free acid is liberated by addition of H_2SO_4 and extracted into ethyl ether. The ether is washed with water until free from other acids and the **I** is determined by titration in ethanol with KOH, with *m*-cresol purple as indicator. By this method 47·0 and 47·3% of **I** were recovered from a sample known to contain 47·2%. G. P. Cook

213. **Rapid identification of elastomers and their additives.** B. Cleverley and R. Herrmann (D.S.I.R., Wellington, New Zealand). *J. Appl. Chem.*, 1960, **10** (5), 192–195.—Elastomers can be identified from the i.r. spectra of their pyrolysates if they are pyrolysed under controlled conditions and interfering substances removed by fractional distillation. Identification of additives separated by fractional sublimation or distillation is possible in many cases. The spectra of the pyrolysates of nine common elastomers are illustrated. G. P. Cook

214. **Quantitative analysis of rubber ingredients. V. Simple combustion tube for the semi-micro determination of sulphur in vulcanisates.** K. Hummel and H. H. Bertram (Kautschukinst., Tech. Hochschule, Hannover, Germany). *Kautsch. u. Gummi*, 1960, **13** (5), WT132–134.—The combustion tube described is packed with a mixture of vanadium pentoxide and quartz (2:1) and, after insertion of the sample, heated to 800° to 1000°. The resulting H_2SO_4 is absorbed in H_2O_2 and titrated complexometrically (*cf.* Bauminger, *Anal. Abstr.*, 1957, **4**, 3389). The rubber-combined sulphur and sulphur in some inorganic compounds, *e.g.*, ZnS, can be determined by this combustion method. Results showing the accuracy of the method for natural rubber vulcanisates of known sulphur contents are given. The sulphur in alkaline-earth-metal sulphates is determined by adding 1 g of a catalyst mixture of vanadium pentoxide and zinc oxide (4:1) to the sample in the boat (*cf.* Bauminger, *Analyst*, 1956, **81**, 12). S. R. P.

215. **Practical identification of some condensed tannins in leather by chromatographic and photometric methods.** D. G. Roux (Leath. Ind. Res. Inst., Grahamstown, S. Africa). *J. Amer. Leath. Chem. Ass.*, 1960, **55** (5), 272–284.—The leather sample is powdered and extracted with acetone - water (1:1) for 2 hr. The soln. is filtered and the filtrate evaporated to dryness *in vacuo*. The extract (3 to 10 mg) is dissolved in 3 to 5 drops of methanol and applied to Whatman No. 1 paper, and developed with water-satd. s-butyl alcohol or n-butanol - acetic acid - water (4:1:5) in one direction and then with 2% acetic acid in the other direction. The paper may be sprayed with diazotised benzidine reagent, vanillin - toluene-*p*-sulphonic acid reagent

moniacal $AgNO_3$ soln., or $(NH_4)_2SO_4.Fe_2(SO_4)_3$ n. The anthocyanidins are recognised by their lorations and by their light-absorption character-ics. C. A. SLATER

216. **Paper electrophoresis and chemical analysis extracts of steerhide corium and of whole steer-de.** C. Deasy (Tanners' Counc. Lab., Univ. of ncinnati, Ohio, U.S.A.). *J. Amer. Leath. Chem. s.*, 1960, **55** (5), 258–271.—Methods of prep. of erhide-corium extracts have been described ewhere (*Ibid.*, 1959, **54**, 503). Whole-steerhide tracts are made from steerhide from a freshly ughtered steer after freezing for one week. The le is cut into squares and extracted successively th 5% NaCl soln., 0·1 M citrate buffer (pH 3·6), M Na_2HPO_4, half-saturated $Ca(OH)_2$ soln. and % $CaCl_2$ soln. Extracts are concentrated by lysis. Glycoproteins are determined by a modi-l Grönwall and Köiw procedure (*Scand. J. Clin. b. Invest.*, 1952, **4**, 244). Results show that ium extracts contain little or no glycoprotein, ilst whole hide contains large quantities. C. A. SLATER

See also Abstracts—**18**, ^2H in org. compounds. **22**, in mineral-oil dispersions. **122**, Cl in tetra-n-tyl titanate. **173**, Analysis of dithiocarbamates. **1**, Detection of glucose. **267**, Chromatography triterpenoids. **283**, Determination of deoxy-gars. **308**, Determination of amylose and ylopectin. **334**, Aldehydes in air. **335**, Toluene l styrene in air. **360**, Recording analyser for . acids. **361**, Fractionation of org. acids. **372**, romatographic determination of ^{14}C-labelled cylic acid. **377**, Response of thermal-conduc-ity detectors to org. compounds. **379**, Gas romatography of alcohols.

4.—BIOCHEMISTRY
INCLUDING DRUGS, FOOD, SANITATION, AGRICULTURE

Biological fluids, animal and vegetable tissues

217. **Desalting of urine by electrodialysis.** T. ood (Biochem. Dept., Univ. of Sydney, New South lles). *Nature*, 1960, **186**, 634–635.—An electro-lyser described by Wood (*Biochem. J.*, 1956, **62**,) for the desalting of tissue extracts has been dified and used by Smith ("Chromatographic hniques, Clinical and Biochemical Applications," inemann, London, 1958) for desalting urine, but siderable losses of amino acids were reported. s now recommended that a "desalting curve" be tted (cell resistance *vs.* time) so that the electro-ysis can be stopped at a point at which most of salt has been removed, but little or no amino-l lost. G. S. ROBERTS

218. **Effects of haemolysis on serum electrolyte ues.** A. Mather and N. R. Mackie (Res. Dept., morial Hosp., Wilmington, Del., U.S.A.). *Clin. m.*, 1960, **6** (3), 223–227.—Correction of the assium and phosphate found in freshly separated molysed sera for the contribution of the cells y be made with reasonable accuracy if the am haemoglobin is determined. The observed ue for K (milli-equiv. per litre) — 3·3 × serum moglobin (g per 100 ml) gives the corrected value for K. The correction for phosphate is ordinarily not significant, but may be made by deducting one third of the serum haemoglobin from the mg of P per 100 ml found. H. F. W. KIRKPATRICK

219. **Automatic method for the determination of carbon dioxide in blood plasma.** L. T. Skaggs, jun. (Dept. of Medicine and Surgery, Veterans Admin. Hosp., Cleveland, Ohio). *Amer. J. Clin. Path.*, 1960, **33** (2), 181–185.—An AutoAnalyser is employed to measure the colour change produced in a phenolphthalein soln. by CO_2. D. E. EVANS

220. **Contemporary carbon-14. The *p*-cymene method.** F. N. Hayes, E. Hansbury and V. N. Kerr (Los Alamos Sci. Lab., Univ. of California, N. Mex., U.S.A.). *Anal. Chem.*, 1960, **32** (6), 617–620.—Carbon-14 in nature is being investigated on a world-wide scale and details are given of the sampling, chemical conversions and liquid scintillation counting procedures used, special attention being paid to the use of *p*-cymene from natural terpene sources for the liquid scintillation method. K. A. PROCTOR

221. **Combined potentiometric determination of serum bicarbonate and chloride.** M. Rockenmacher (Clin. Lab., Dept. of Pathology, Los Angeles County Harbor General Hospital, Torrance, Calif.). *Amer. J. Clin. Path.*, 1960, **33** (4), 349–354.—As little as 0·2 ml of serum is sufficient for this titrimetric method, the results of which compare well with those of the Van Slyke and Whitehorn methods. D. E. EVANS

222. **Determination of sodium and potassium in biological fluids with the dual-channel ultra-micro flame photometer.** P. A. Bott (Dept. of Physiol. Chem., Woman's Med. Coll. of Pennsylvania, Philadelphia, U.S.A.). *Anal. Biochem.*, 1960, **1** (1), 17–22.—A modification of the instrument designed by Ramsay *et al.* (*J. Exp. Biol.*, 1953, **30**, 1) is described. As little as 2 $\mu\mu$moles of Na and K can be determined simultaneously in 0·2 μl of sample. H. F. W. KIRKPATRICK

223. **Analysis for potassium, sodium, chloride and water in a 2-μl sample of extracellular fluid.** A. Ames, III, and F. B. Nesbett (Harvard Medical School, Boston, Mass.). *Anal. Biochem.*, 1960, **1** (1), 1–7.—The samples (2 to 8 μl) are weighed into small stoppered tubes, dried for 4 hr. at 105°, and re-weighed to give the water content. The extracting soln. (0·220 millimole of $AgNO_3$ and 5 millimoles of Li_2SO_4 per litre of 0·1 N HNO_3) is added in a vol. corresponding to 1000 times the water content of the sample. The Cl^- are determined by measuring the residual Ag^+ spectrophotometrically with the rhodanine reagent of Lowry *et al.* (*J. Biol. Chem.*, 1954, **207**, 1). The K and Na are determined by flame photometry in a specially designed photometer, the Li^+ in the extraction soln. serving as internal standard. The accuracy and reproducibility are better than 2%. R. A. BRENAN

224. **Determination of free magnesium ions in body fluids. Improved methods for free calcium ions, total calcium and total magnesium.** M. Walser (The Johns Hopkins Univ. Sch. of Med., Baltimore, Md., U.S.A.). *Anal. Chem.*, 1960, **32** (6), 711–717.—The colour change on the addition of Eriochrome black T to a protein-free sample of body fluid, buffered with tri(hydroxymethyl)-methylamine, gives an accurate estimate of the free Mg^{2+} concn. At constant pH and constant

ionic strength, the reciprocal of the extinction difference is a linear function of the reciprocal of the Mg^{2+} concn. Heavy-metal ions must be removed before the analysis; other cations present in body fluids do not interfere. A similar procedure is used for free Ca^{2+}, with murexide as indicator. The sum of total Ca and total Mg is determined by titration with EDTA (disodium salt) soln. at pH 10, with Eriochrome black T as indicator. Total Ca is determined by titration in strongly alkaline soln., with Cal-Red as indicator (Bachra *et al.*, *Clin. Chem.*, 1958, **4**, 107). A. R. ROGERS

225. **Micro calcium method accurate in the presence of magnesium and phosphate.** T. B. Coolidge (Dept. of Biochem., Univ., Chicago, Ill.). *Anal. Biochem.*, 1960, **1** (1), 93–95.—Serum (0·5 ml) is pipetted into a Pyrex-glass tube (100 mm × 13 mm) and dried at 110°. A mixture (0·2 ml) of equal parts of HNO_3 and 70% $HClO_3$ is added and the tube is placed for 5 min. at a depth of 20 mm, for 15 min. at a depth of 40 mm, and for 30 min. at a depth of 98 mm in an aluminium block heated to 180° and provided with suitable holes and plugs to give the required depths. To the ash is added water (0·5 ml), fresh aq. dilution (1 : 5) of stock 2% calcein soln. in 0·25 N NaOH (2 drops), and 2 N CO_2-free NaOH (0·4 ml). About 0·5 ml (measured to 0·1% accuracy) of 0·004 M EDTA, prepared by dilution of 0·04 M EDTA in 0·081 N NaOH, is added from a syringe pipette, and the mixture is titrated with 0·100 M $CaCl_2$ from a micro-burette, with stirring by filtered air. R. A. BRENAN

226. **Photo-electric complexan titration of calcium and magnesium in serum.** M. Kortüm (Inst. f. Phys. Chem., Univ. Tübingen, Germany). *Klin. Wochschr.*, 1960, **38** (9), 452–457.—The following procedures are proposed. *Calcium*—Mix in a cell 0·0025 M EDTA (disodium salt) (1·0 ml), N NaOH (1·5 ml), 5% Na_2S soln. (0·15 ml), serum (1·0 ml), 0·1% aq. murexide soln. (0·15 ml) and H_2O (3·5 ml) in that order. Place the cell in the absorptiometer and set the extinction to 0·1 to 0·3 at 490 mμ. Add 0·0025 M Ca soln. dropwise from a micro-burette, taking readings after each addition. The extinction at first remains approx. constant, then rises. The end-point is reached when the addition of a drop of titrant produces a permanent increase in the extinction. Then mg of Ca per 100 ml of serum = (2·0 — no. of ml of Ca soln. added) × 10. *Magnesium*—Mix in a cell 0·0025 M EDTA (disodium salt) (2·0 ml), 2·5% aq. NH_3 soln. (1·5 ml), 5% Na_2S soln. (0·2 ml), serum (1·0 ml), 0·1% aq. catechol violet soln. (0·15 ml) and H_2O (3·5 ml) in that order. After 2 min., place the cell in the absorptiometer and set the extinction to approx. 0·8 at 620 mμ. Titrate dropwise as described above. Then mg of Mg per 100 ml of serum = (no. of ml of Ca soln. added in the murexide titration — ml of Ca soln. added in the catechol violet titration) × 6·1. H. F. W. KIRKPATRICK

227. **Complexometric determination of calcium and magnesium in urine.** S. I. Pekhtereva (Lenin Inst. of Med., Moscow). *Lab. Delo*, 1960, **6** (3), 17–20.—*Procedure*—To 5 ml of urine add 5 ml of 0·2 N Na oxalate, mix and set aside for 1 hr. at room temp., then filter. To one flask (*a*) add 1 ml of urine and 1 ml of water, and to another (*b*) 2 ml of filtrate. Then to each flask add 1 ml of 0·03% ethanolic methyl red soln., 1 ml of 0·1% ethanolic Chromogen black ET-00 (C.I. Mordant Black 11) soln. and 0·5 ml of buffer soln. (20 g of NH_4Cl and 10 ml of 25% aq. NH_3 in 1 litre of water). Titrate with 0·01 N EDTA (disodium salt) dropwise, with vigorous shaking after each addition, to a colour change from violet - red to green. The amount of EDTA used in (*a*) corresponds to the sum of Ca and Mg and the amount used in (*b*) to Mg alone; Na_2HPO_4 does not interfere, and the errors are ≯3%. A. BURWOOD-SMITH

228. **Wet digestion of plant material gives low boron values.** J. T. Hatcher (Soil and Water Conservation Res. Div., U.S. Dept. of Agric., Riverside, Calif.). *Anal. Chem.*, 1960, **32** (6), 726.—Results on 12 samples show that the wet-digestion procedure (*cf. U.S. Dept. Agric., Agric. Handb.*, 1954, **60**, 129, 135) is not satisfactory. Serious loss of boric acid occurs by volatilisation from the acid soln. during the digestion. A. R. ROGERS

229. **Determination of silicic acid in biological material.** H. Baumann (Silikose-Forschungsinst., Bergbau-Berufsgenossenschaft, Bochum, Germany). *Hoppe-Seyl. Z.*, 1960, **319**, 38–51.—A colorimetric method is described for the determination of silicic acid by the reduction of the molybdosilicate complex with a mixture of 1-amino-2-naphthol-4-sulphonic acid and ascorbic acid. Other reducing substances, PO_4^{3-} and Fe do not interfere. Protein is removed by combustion or ultrafiltration. A 3-ml sample is used and one operator can carry out 40 to 50 analyses in 8 hr. Much lower results have been obtained with this procedure than have been found with other techniques. Bovine blood or plasma has a silicic acid content of 2·2 μg per ml. This is not bound to protein or other substances of high mol. wt. F. POWELL

230. **Micro-method for estimating inorganic phosphorus in serum or plasma.** J. F. Johnston (Biochem. Dept., Royal Hospital for Sick Children, Glasgow). *J. Med. Lab. Technol.*, 1960, **17** (1), 25–27.—This is a modification, for application to micro amounts, of the method of Bell and Doisy (*J. Biol. Chem.*, 1920, **44**, 55). R. A. BRENAN

231. **Determination of phosphorus, radio-phosphorus and chromic oxide in faeces with a solution obtained by perchloric acid oxidation.** Ichiro Yoshihara and Akio Sugisaki (Agric. Fac., Niigata Univ., Japan). *J. Agric. Chem. Soc. Japan*, 1959, **33** (1), 27–31.—Chromic oxide and ^{32}P were used as tracers for the study of absorption and excretion of P in rabbits, and the separate determination of Cr, ^{32}P and total P in faeces was carried out as follows. The sample (≃1·2 g) is heated with 60% $HClO_4$ soln. (20 ml) for 30 min., then cooled and treated with H_2O_2 (10 drops). The soln. is heated again, mixed with water (80 ml), filtered and diluted to 200 ml. This soln. is used for the determination of total P by Allen's method (*Biochem. J.*, 1940, **34**, 858), of ^{32}P by counting after pptn. with magnesia mixture, and of Cr by colorimetry. S. NATORI

232. **Determination of chromic oxide and barium sulphate used as a trace material for the movement of intestinal contents.** Ichiro Yoshihara (Agric. Fac., Niigata Univ., Japan). *J. Agric. Chem. Soc. Japan*, 1959, **33** (2), 138–141.—The conditions for the separate determination of $BaSO_4$ (**I**) and Cr_2O_3 (**II**) were examined, and the determination of **I** by the isotope dilution method and **II** by titration was improved and applied to the determination of **I** and **II** in faeces. S. NATORI

233. **Spectrographic determination of zinc in plant terial ash using the 2138·6-A spectrum line.** A. asheim and D. J. Eve (Nat. Phys. Res. Lab., African Council for Sci. & Ind. Res., Pretoria). *S. Afr. Chem. Inst.*, 1960, **13** (1), 7–12.—In the thod described, Ilford "Q1" u.v.-sensitive plates re used, and modifications were made to earlier ocedures to minimise errors. A Stallwood-type jet was used round the arc to overcome the ficulty of self-absorption. A constant-current rce (Van der Walt, *Rev. Sci. Instrum.*, 1956, **27**, 7) minimised variations in emission. The sample s diluted (1:9) with a buffer mixture of 10% of SO_4 and 40% of $CaCO_3$ in graphite to reduce the luence of variation in the main constituents of sample. The method is fairly rapid, apart m the ashing, and is sufficiently accurate for tritional studies on trace elements. The error is 5·4%. A. M. Spratt

234. **Specific detection of glucose on paper chromograms.** M. R. J. Salton (Dept. of Bacteriology, niv. Calif., Berkeley, U.S.A.). *Nature*, 1960, **186**, 6–967.—Glucose may be detected and distinguished from other monosaccharides by spraying e dried chromatogram with a glucose oxidase dicator reagent ("Glucostat," Worthington Biomical Corp.). When acid solvents, such as butanol - acetic acid, have been used, the dried per should be steamed for 5 to 10 min. before raying. Glucose develops a pink - brown spot room temp., and may be detected in amounts of to 20 mμg, or less. The coloured complex can t be eluted for quantitative determination. ter detection of glucose, other monosaccharides ay be detected by a further spraying with aniline thalate. D. G. Moss

235. **Determination of alcohol in blood by gas romatography.** B. Chundela and J. Janák ab. Toxicol. and Forensic Med., Charles' Univ., ague). *Soudní Lékařství*, 1959, **4** (10), 145–150.—2% aq. soln. of n-butanol (0·25 ml) is added to e sample (1 ml) and 0·1 ml of the mixture is aced by means of a syringe on the column. e column consists of 25% of diglycerol on Stermol (0·2 to 0·4 mm mesh) activated for 3 hr. at 0°. A Griffin and George apparatus, type MK II, used, and the n-butanol serves as an internal ndard. The sensitivity is 0·1%. J. Zýka

236. **Identification of corticosterone sulphate in man urine after administration of corticosterone.** R. Pasqualini. *Compt. Rend.*, 1960, **250** (23), 92–3893.—It is shown that sulphate ester is creted within 36 hr. by a normal subject given rticosterone. The ester is separated by two romatograms on Al_2O_3 and identified by chromatography in four solvent systems, synthetic corticone 21-sulphate being run in parallel. The rrent alcohol is similarly identified, after hydrolysis with sulphatase (from *Helix pomatia*), by chromography in three solvent systems, with corticone run in parallel. J. P. Stern

237. **Determination of starch in leaves and other rts of plants.** Kh. N. Pochinok. *Nauch. Trud. ser. Inst. Fiziol. Rast.*, 1959, (20), 59–62; *Ref. Zur., Khim., Biol. Khim.*, 1960, (9), Abstr. No. 1826.—Volumetric and colorimetric methods have en developed. Dissolve the starch by heating th 80% $Ca(NO_3)_2$ soln. and separate it from other bstances by pptn. with iodine. The resulting ppt. starch is quant. oxidised in H_2SO_4 medium with $K_2Cr_2O_7$ to CO_2 and water, which enables it to be determined by a simple volumetric method. From 1 to 10 mg of starch can be determined, with an accuracy of ±1%. The starch pptd. with iodine can also be determined colorimetrically after dissolving the starch - iodine complex in alkali; 0·5 to 5 mg of starch can be determined, with an accuracy of ±5%. C. D. Kopkin

238. **Micro-method for the determination of laevulic acid.** Nai-Siang Jiang and D. P. Groth (Dept. of Pharmacol. and Biochem., Emory Univ., Atlanta, Ga.). *Anal. Biochem.*, 1960, **1** (1), 40–43. – The dinitrophenylhydrazone is formed in benzene soln. in the presence of H_2SO_4, and extracted with 0·5 N NaOH. The colour is measured at 540 mμ. The method can be used to determine 2·5 to 25 μg of laevulic acid. H. F. W. Kirkpatrick

239. **New technique for the determination of galacturonic acid in cerebrospinal fluid.** L. Buscarini (Ist. di Clin. Med. Generale e Terapia Med., Univ. Pavia, Italy). *Boll. Soc. Ital. Biol. Sper.*, 1960, **36** (11), 564–567.—The method is based on the application of the Dische reaction (*J. Biol. Chem.*, 1947, **167**, 189) to the determination of galacturonic acid separated from protein by pptn. with protamine sulphate. *Procedure*—To cerebrospinal fluid (1 ml) add water (4 ml) and a 1% soln. of protamine sulphate (0·3 ml), shake and centrifuge. Dissolve the ppt. in 2 M acetate buffer (pH 5) (3 ml). To the sample soln. (1 ml), to a standard soln. containing 5 mg of glucuronic acid per 100 ml (1 ml), and to a buffer blank (1 ml) add H_2SO_4 (6 ml), drop by drop, with cooling. Heat at 100° for 20 min., allow to cool and measure the developed (non-specific) colour at 535 mμ. To each soln. add a 0·1% ethanolic soln. of carbazole (0·2 ml), set aside for 2 hr. and measure the colour developed at 535 mμ. The increase in colour in the sample soln. is calculated to glucuronic acid; glucuronic acid found × 2·32 = galacturonic acid. Results obtained for normal human subjects were 177 ± 8·70 mg of galacturonic acid per 100 ml. E. C. Apling

240. **Enzymatic determination of hyaluronic acid.** H. Greiling, T. Gunther and T. Eberhard (Physiol.-Chem. Inst., Freien Univ., Berlin). *Hoppe-Seyl. Z.*, 1960, **319**, 161–166.—The u.v. absorption of the disaccharide formed by the action of bacterial hyaluronidase may be used to determine hyaluronic acid. The material to be tested is incubated for 6 hr. at pH 6·4 with a preparation of hyaluronidase from *Staphylococcus aureus* and the extinction is read at 230 mμ. Beer's law is obeyed over the range 20 to 700 μg of hyaluronic acid. Above a mol. wt. of 4000 the method is not influenced by the degree of polymerisation of the hyaluronic acid. The mean error is ±2%. F. Powell

241. **Positive identification of flavanone aglycones by paper chromatography of their alkaline degradation products.** W. J. Dunlap and S. H. Wender (Chem. Dept., Univ. of Oklahoma, Norman, U.S.A.). *J. Chromatography*, 1960, **3** (5), 505–507 (in English).—Flavanones are degraded by heating the aglycone under reflux with a little 30% aq. KOH soln. for 2·5 hr. The mixture is then made acid (pH 4) with dil. H_2SO_4 and extracted with ethyl ether, and the extract is chromatographed. The solvents used are n-butanol - ethanol - aq. NH_3/ ammonium carbonate buffer (40:11:19) and a modification prepared from 1 part of nitromethane

with 2 parts of n-butanol saturated with the ammoniacal buffer. Diazotised sulphanilic acid and *p*-nitroaniline spray reagents are used for the detection of hydroxycinnamic acids and phloroglucinol, but for cinnamic acids possessing only methoxy substituents, and which do not therefore couple, buffered methyl red or 2,6-dichlorophenol-indophenol reagents are useful. The method has been successfully applied to the identification of unknown compounds obtained by hydrolysing the glycosides from citrus fruits. S. M. MARSH

242. **Isolation and identification of lysolecithin from lipid extracts of normal human serum.** E. Gjone, J. F. Berry and D. A. Turner (Biochem. Res. Div., Dept. Med., Sinai Hosp., Baltimore, Md., U.S.A.). *J. Lipid Res.*, 1959, **1**, 66–71.—Lipids were extracted from 15 to 20 ml of serum with 25 vol. of $CHCl_3$ - methanol (2:1). The lipid extract was washed with water and the solvent removed. A soln. of the lipids in $CHCl_3$ was subjected to chromatography on silicic acid. Ethyl ether was used to elute the non-phospholipids, then the column was eluted with $CHCl_3$ - methanol. The phospholipids eluted successively were cephalin, lecithin, sphingomyelin and lysolecithin. The identity of each component was confirmed by paper chromatography, chemical analysis and infra-red absorption spectra. Lysolecithin P represented $\simeq 9\%$ of the total lipid P of serum. NUTR. ABSTR. REV.

243. **Determination of serum glycerides.** L. A. Carlson (King Gustav V Res. Inst., Stockholm). *Acta Soc. Med. Uppsaliensis*, 1959, **64**, 208–213.—Lipids are extracted with $CHCl_3$ - methanol (2:1) from 1 ml of serum and the extract is washed with NaCl soln. The remaining $CHCl_3$ soln. is passed through the column of silicic acid on which the phospholipids are selectively retained. A portion of the $CHCl_3$ percolate is then saponified with ethanolic KOH soln., dil. H_2SO_4 is added and fatty acids and unsaponifiable matter are extracted with ethyl ether; the residual aq. layer is used for the determination of glycerol by the method of Carlson and Wadström (*Anal. Abstr.*, 1959, **6**, 4083).
NUTR. ABSTR. REV.

244. **Seromucoid determination: variables and modifications.** T. M. Asher and G. R. Cooper (Communicable Diseases Centre, Public Health Service, Atlanta, Ga., U.S.A.). *Clin. Chem.*, 1960, **6** (3), 189–198.—The method of Winzler ("Methods of Biochemical Analysis," 2nd Ed., New York, 1955, p. 279) is investigated. A modified technique is described for making the test more sensitive. H. F. W. KIRKPATRICK

245. **Enzymatic spectrophotometric method for the determination of uric acid.** L. Liddle, J. E. Seegmiller and L. Laster (Nat. Inst. Health, Bethesda, Md., U.S.A.). *J. Lab. Clin. Med.*, 1959, **54** (6), 903–913.—A detailed description of the uricase method (*cf.* Feichtmeir and Wrenn, *Amer. J. Clin. Path.*, 1955, **25**, 833) and an evaluation of the method in 4000 analyses during clinical studies of uric acid metabolism are given. The specificity, reproducibility and accuracy, and results in relation to those given by other methods, are discussed.
W. H. C. SHAW

246. **Effect of orally administered quinine and quinidine on apparent values for urinary catecholamines.** S. M. Sax, H. E. Waxman, J. H. Aarons and H. J. Lynch (Dept. of Path., W. Pennsylvania Hosp., Pittsburgh, U.S.A.). *Clin. Chem.*, 1960, **6** (2), 168–175.—Oral administration of quinine or quinidine yields urinary derivatives, possibly carbostyrils, that cause an apparent increase in urinary catecholamines.
H. F. W. KIRKPATRICK

247. **Determination of creatinine in small quantities of plasma.** E. Martinez and P. D. Doolan (Clin. Invest. Centre, U.S. Naval Hosp., Oakland, Calif.). *Clin. Chem.*, 1960, **6** (3), 233–242.—A modification of the method of Van Pilsum *et al.* (*J. Biol. Chem.*, 1956, **222**, 225) is described and is recommended when accuracy is the primary requirement. For routine use a modification of the alkaline picrate method is proposed.
H. F. W. KIRKPATRICK

248. **Determination of choline in blood by antimony chloride in ethereal solution.** H. Wachsmuth and R. Denissen (Lab. de Chim. et de Biochim., Hôpital Stuivenberg, Anvers). *J. Pharm. Belg.*, 1959, **14** (3–4), 141–144.—*Procedure*—Treat 3 to 4 ml of blood with Bloor's mixture. Evaporate the alcoholic soln. and re-dissolve the residue in 1 ml of light petroleum. Add 8 ml of acetone and 2 to 3 drops of satd. ethanolic $MgCl_2$ soln. to precipitate the phosphatides. Treat the ppt. with satd. $Ba(OH)_2$ soln. and heat on a boiling-water bath for 2 hr. Acidify with N H_2SO_4, then make alkaline with 0·05 N NaOH, and neutralise with 0·1 N HCl to the phenolphthalein end-point. Centrifuge, evaporate an aliquot to dryness at $\gtrsim 40°$, re-dissolve the residue in ethanol and reduce the vol. to 0·2 to 0·3 ml in a centrifuge tube. Add SbI_3 soln. (make a suspension of 0·2 g of SbI_3 in 5 ml of ethanol; after 3 min. add 15 ml of peroxide-free ethyl ether and filter), dropwise, and make up to 7 to 8 ml with ether. Centrifuge the yellow ppt. in a closed tube, decant off the ether, and wash the ppt. twice with ether. Dissolve the ppt. in 1 ml of 30% NaOH soln., dilute to $\simeq 100$ ml, acidify slightly with H_3PO_4 and add 20 ml of bromine water. Remove the excess of Br by boiling, add 2 to 3 drops of 5% Na salicylate soln., acidify with H_3PO_4, and add solid KI. Titrate the liberated iodine with 0·01 N $Na_2S_2O_3$ (1 ml $\equiv$ 0·0871 mg of choline chloride). Results are compared with those obtained with ammonium reineckate. R. A. HENDEY

249. **Sensitivity of the fluorescence test for amino-acids.** J. Opieńska-Blauth, M. Sanecka and M. Chareziński (Biochem. Dept., Med. Acad., Lublin, Poland). *J. Chromatography*, 1960, **3** (5), 415–424 (in English).—The sensitivities of the ordinary u.v. fluorescence test, the sensitised u.v. test (sensitising agent Na 1,2-naphthaquinone-4-sulphonate soln.) and the ninhydrin and isatin tests have been compared for 25 amino-acids. The sensitised u.v. test was ten times as sensitive as the ordinary u.v. test, except with asparagine, lysine and tryptophan. The best results are obtained by heating the chromatogram at 110° to 120° for 10 min. The effect of developing the chromatogram with phenol - water (7:3) and n-propanol - water (7:3) was to reduce the sensitivity owing to losses during migration, particularly for amino-acids with high R_F values. Amino-acids in biological fluids such as urine and blood serum can be identified by applying the u.v. and ninhydrin tests successively, provided that the amount of sample applied is not less than 100 μl. S. M. MARSH

50. **Determination of amino-acids with dithionite-ıced ninhydrin.** H. Stegemann (Max-Planck-ellschaft, Göttingen, Germany). *Hoppe-Seyl.* 1960, **319**, 102–109.—A procedure is described the determination of amino nitrogen by means ninhydrin partially reduced with dithionite. e presence of NaF and a high concn. of acetate fer in the reagent make it possible to use soln. taining air. The optimum range is 1 to 3 μg N per ml. The accuracy in this range is $\pm 2\%$; r's law is obeyed up to 10 μg. The colour is ble for 1 hr. and it is claimed that it is possible nalyse more than 1000 samples per day.

F. Powell

51. **Determination of neutral amino-acids by tition chromatography on silica gel.** Makoto ndatsu and Hiroshi Naito (Fac. of Agric., Univ., kyo). *J. Agric. Chem. Soc. Japan*, 1959, **33** (3), –174.—The separation of *N*-acetamido-acids by omatography on silica gel was investigated. e quality of the gel, *e.g.*, its purity, particle size l water content, especially of structural water, med to affect significantly the recovery of amino-ds. A mixture of acetylated amino-acids was ted with ethyl acetate and then with $CHCl_3$ - anol containing an increasing proportion of anol. The determination of valine in the pres-e of methionine was carried out after performic d oxidation followed by acetylation and chrom-graphy. Amino-acid components of casein and letal muscular protein of albino rats were ermined by this method. S. Natori

52. **Electrophoretic separation of some amino-ds from their copper complexes.** M. Szwaj and Kański (Biochem. Dept., Med. Acad., Lublin, and). *J. Chromatography*, 1960, **3** (5), 425–430 English).—Complete separation of amino-acid - per complexes into three groups, acid, neutral alkaline, and separation of a given complex m the free amino-acid has been achieved by er electrophoresis in a borate - NaOH buffer, 10, μ 0·05, at a potential gradient of 6·6 to 2 V per cm. The sample mixture is applied f way along the paper strip and electrophoresis arried out for 1·5 hr. Ninhydrin is used as a ting agent for amino-acids and a 0·1% soln. of io-oxamide in methanol for the copper com-xes. Results are detailed for 9 amino-acids and ir complexes. The composition of the complex be determined by precipitating the Cu with ; and chromatographing the liberated acid.

S. M. Marsh

53. **Quantitative determination of 5-hydroxy-)tophan, 5-hydroxytryptamine and 5-hydroxy-)l-3-ylacetic acid by paper electrophoresis.** A. casona, F. Unterharnscheidt, J. Cervos-Navarro W. Geller (Inst. f. Neuropathol., Univ. Bonn, many). *Klin. Wochschr.*, 1960, **38** (9), 457–460. n barbitone buffer (pH 8·6), acetate buffer 5·05) and phosphate buffer (pH 7·0), 5-roxytryptamine travels towards the cathode, ydroxyindol-3-ylacetic acid towards the anode, 5-hydroxytryptophan remains stationary. The ee substances are therefore separable and can be ermined by spraying the paper with *p*-dimethyl-nobenzaldehyde soln. in n-butanol (1:4) and ting at 60° for 20 min. to produce a colour which easured planimetrically on the paper at 540 mμ. procedure is described.

H. F. W. Kirkpatrick

254. **Reduction of cystine as a basis for a simple amido-group determination for all proteins.** H. Stegemann (Max-Planck-Gesellschaft, Göttingen). *Hoppe-Seyl. Z.*, 1959, **315**, 137–140.—Performic acid oxidation of cystine before the determination of amide nitrogen in proteins (*cf. Anal. Abstr.*, 1959, **6**, 4097) is not satisfactory for proteins rich in tryptophan. Reduction by Na thioglycollate or Na_2S is satisfactory for all proteins. The reduction of the cystine, alkaline hydrolysis of the amido-group, and diffusion of the NH_3 formed into H_2SO_4 are all carried out in one step. The actual working time is no longer than for a simple estimation of amido-groups, but 65 hr. is required for the complete diffusion of the NH_3. F. Powell

255. **Chromatographic determination of cysteic acid.** S. L. Bandemer and R. J. Evans (Dept. of Agric. Chem., Michigan State Univ., East Lansing, U.S.A.). *J. Chromatography*, 1960, **3** (5), 431–433 (in English).—The difficulties caused by variations in the volume of eluate fractions obtained in the determination of cysteic acid with ninhydrin, after its chromatographic separation on a resin column, have been overcome by the use of buffered soln. of 2 N NaOH saturated with Na citrate to produce the pH 5 required for maximum colour development. By using collector tubes calibrated to 10 ml and by constructing a standard cysteic acid curve the calculation of cysteic acid concn. has been simplified.

S. M. Marsh

256 **Technique for the identification of peptides on paper by means of chlorine and o-tolidine.** O. J. Schmid (Physiol.-Chem. Univ.-Inst., Hamburg, Germany). *J. Chromatography*, 1960, **3**(5), 499–500 (in German).—The paper chromatogram is sprayed with acetone - 30% ethanol (1:1) and suspended in freshly generated chlorine gas for 3 to 10 min. It is then transferred to a second vessel containing gaseous NH_3 and suspended there for 5 to 10 sec. The edge of the paper is tested by spraying with tolidine reagent (prepared from a satd. soln. of *o*-tolidine in 2% acetic acid with an equal vol. of 0·05 M KI) and if the background gives a blue colour the treatment with NH_3 is repeated. The whole chromatogram is then sprayed with the reagent. Amino-acids or peptides give blue - black or brown - yellow flecks, which can be photographed or photo-copied before they fade. S. M. Marsh

257. **Combined agar gel - paper electrophoresis.** B. Zak, F. Volini, J. Briski and L. A. Williams (Dept. of Pathology, Wayne State Univ. Coll. of Med., Detroit, Michigan). *Amer. J. Clin. Path.*, 1960, **33** (1), 75–82.—A method of agar-gel electrophoresis is described, whereby it is possible to evaluate the concn. of stained proteins in the separated zones; 25 to 45 min. is required for the complete separation of 5 or more protein zones. Resolution of serum proteins or haemoglobins in this open system is similar to that obtained with a closed system. Elution recoveries of separated serum proteins are almost identical with those of the Spinco procedure. Wet paper is laid on a glass plate and a boundary wall is formed of glass rods, to contain the molten agar. After electrophoresis, the gel - paper combination is dried and stained. Drying concentrates the protein bands down on to the surface of the paper, rather than into its substance, thus tending to give sharper definition of zones.

D. E. Evans

258. **Automatic determination of serum proteins.** J. F. Failing, jun., M. W. Buckley and B. Zak (Dept. of Pathology, Wayne County General Hospital, Eloise, Michigan). *Amer. J. Clin. Path.*, 1960, **33** (1), 83–88.—Two absorptiometric methods are described. In the first, the biuret reaction is used for the determination of total serum proteins and, in the second, the selective reaction of methyl orange with serum albumin is used for the determination of this protein in the presence of other serum proteins. Concentration - transmittance curves are compiled for the AutoAnalyser, with standard protein solutions. In general the results compare well with those obtained by manual biuret-salt fractionation and manual methyl orange absorption. The values obtained by the automatic method show a slight compression of the range (high values less high, low values less low) compared with the values obtained manually. D. E. Evans

259. **Electrophoresis of soluble proteins in agar gel.** A. T. Ilkov and T. Nikolov. *Voprosy Med. Khim.*, 1959, **5** (5), 388–392; *Ref. Zhur., Khim., Biol. Khim.*, 1960, (7), Abstr. No. 8788.—The electrophoresis on agar of blood-serum proteins has been carried out with a potential gradient of 2·5 V per cm in a barbitone buffer of pH 8·6, with μ 0·05, and current 10 to 13 mA per sq. cm of cross-section of the agar. Under these conditions the serum proteins are separated by 12 to 13 cm in 4 to 4·5 hr. After the electrophoresis, the plate was twice immersed in a bath of 0·2 N acetic acid for 8 to 12 hr. and then dried in a thermostat at 37°. The dried plates were coloured for 5 hr. in a soln. of Amido black 10 B (C.I. Acid Black 1), and the background decolorised by immersion for 1 to 2 min. in 2% acetic acid containing 2·5% of glycerol. The dried plates were studied densitometrically. In the study by this method of human and animal blood serum, the presence of β_1- and β_2-globulins was noted, in addition to the peaks of albumin and α_1-, α_2-, β- and γ-globulins obtained by paper electrophoresis. In a large number of cases with human serum and in all cases with rabbit serum, an additional fraction, α_3-, was observed between the α_2- and β_1-fractions. Even more protein fractions were observed with dog sera. In some sera the β_2- and γ-globulin fractions were divided into sub-fractions. The described method has been used to determine the soluble proteins of tissues and the urease activity of the separate protein fractions of extracts of the mucous membrane of the stomach, urease preparations from soya, etc.

C. D. Kopkin

260. **Separation of human serum lipoproteins by conventional descending and centrifugally accelerated paper chromatography.** H. J. McDonald, L. J. Banaszak and J. Q. Kissane (Dept. of Biochem., Loyola Univ., Chicago, Ill.). *Anal. Biochem.*, 1960, **1** (1), 44–59.—The paper chromatography of lipids, including the centrifugally accelerated technique (McDonald *et al.*, *Anal. Chem.*, 1959, **31**, 825), is described. Correlation was established between lipoprotein fractions obtained by these methods and by ultracentrifugation.

H. F. W. Kirkpatrick

261. **Separation of the α- and β-chains of globins by means of starch-gel electrophoresis.** C. J. Muller (Dept. of Paediatrics, Univ. of Groningen, Holland). *Nature*, 1960, **186**, 643.—Human adult and foetal globin has been separated in chains by the starch-gel electrophoresis method of Smithies (*Biochem. J.*, 1955, **61**, 629). The α-chains of the two types of globin have equal mobilities, whereas the β-chains have different mobilities.

G. S. Roberts

262. **Improved method for the determination of haemoglobin A_2 by starch-gel electrophoresis.** C. A. J. Goldberg and A. C. Ross (Wm. Pepper Lab. of Clin. Med., Univ. of Pennsylvania, Philadelphia, U.S.A.). *Clin. Chem.*, 1960, **6** (3), 254–262.—Modifications introduced into a method previously published (Goldberg, *Ibid.*, 1958, **4**, 484) improve the accuracy. H. F. W. Kirkpatrick

263. **A new electrophoretic medium and its application to the resolution of human haemoglobin variants.** E. R. Huehns and A. O. Jakubovic (Dept. of Biochem., University College, London). *Nature*, 1960, **186**, 729–730.—The separation of human haemoglobin variants by gel-phase ion-exchange electrophoresis is investigated. Carboxymethylcellulose gel is the supporting medium and electrophoresis is carried out in a sodium phosphate buffer (0·17 to 0·04 M Na^+). The best over-all separation, including the separation of haemoglobins A and F, is achieved with a Na^+ concn. of 0·07 M. E. C. Butterworth

264. **Far ultra-violet direct photometry applied to electrophoresis of nucleic acid.** N. Ressler (Dept. of Med., Wayne County General Hospital, Eloise, Mich., U.S.A.). *Naturwissenschaften*, 1960, **47** (10), 228–229 (in English).—Zone electrophoresis is performed in the usual way in a film of transparent semi-fluid or thixotropic buffer layered on a quartz plate. At intervals during the course of the electrophoresis, light from a monochromator is passed through the film on to a photographic plate. Alternatively, a spectrophotometer photocell can be used. The migration of protein and nucleic acid can be observed at 200 mμ, then by changing the wavelength to 260 mμ, at which protein does not absorb, the nucleic acid alone can be observed. The method has been used to study the interaction between nucleic acid and protein during migration.

R. A. Brenan

265. **Paper chromatography of nucleic acid derivatives.** R. C. Ramos Barreto. *Rev. Quim. Ind., Rio de Janeiro*, 1960, **29**, 15–16.—Examples of paper-chromatographic separations of nuclei acid derivatives, including ^{32}P-labelled components are described. L. A. O'Neill

266. **Determination of cholesterol with specia reference to the use of toluene-*p*-sulphonic acid.** L. A. Wright, D. B. Tonks and R. H. Allen (Dept. c Nat. Health and Welfare, Ottawa, Ont., Canada). *Clin. Chem.*, 1960, **6** (3), 243–253.—The method c Pearson *et al.* (*Anal. Chem.*, 1953, **25**, 813) is com pared with six alternative methods and is found t be rapid, to yield comparable results, and to b suitable for routine purposes. The explosiv hazard is not confirmed, but sulphosalicylic aci may be substituted for the toluene-*p*-sulphonic aci if desired. H. F. W. Kirkpatrick

267. **Paper chromatography of steroids an triterpenoids. II. Paper chromatography of steroli** Itsuo Nishioka (Pharm. Inst., Med. Fac., Kyush Univ., Katakasu, Fukuoka). *J. Pharm. Soc Japan*, 1959, **79** (11), 1453–1456.—Thirteen sterol

e separated by reversed-phase paper chroma-raphy, with liquid paraffin as the stationary se and a polar solvent system (*e.g.*, methanol-er, propanol - methanol - water or 2-ethoxy - anol - propanol - methanol - water) as the mobile se. The relationship between structure and the value of the sterols is discussed.

II. Paper chromatography of triterpenoids. io Nishioka. *Ibid.*, 1959, **79** (11), 1457–1461.— paper chromatography of triterpenes and erpenoid saponins was examined with a number olvent systems on paper saturated with water, id paraffin or methyl stearate. The relationship ween chemical structure and R_F value is dis-sed. S. Natori

68. **Qualitative, quantitative and preparative omatography of steroids on fully acetylated paper. Quantitative chromatography of Δ^4- and $\Delta^{1,4}$- xosteroids.** J. Hartel, A. Bol Raap and F. J. ter (Central Lab. T.N.O., Delft, The Nether-ls). *J. Chromatography*, 1960, **3** (5), 482–487 English).—The method involves chromatography benzene - methanol - water (4:4:1) on fully tylated paper, the steroid spots being subse-ntly cut out, eluted and determined spectro-tometrically. Exact determination is possible ause of the absence of impregnating agent and ht adsorption of the steroids on the paper. ults are presented for cholest-4-ene-3-one, gesterone, testosterone, cortisol and prednisolone. S. M. Marsh

69. **Glass-fibre-paper chromatography of adrenal gonadal steroids.** J. F. Dingman, M. C. Staub, Gaitan and G. Bazzano (Endocrine Unit, Univ. of Med., New Orleans, La., U.S.A.). *Clin. m.*, 1960, **6** (3), 228–232.—Glass-fibre paper X-934-A.H. (H. Reeve Angel & Co.) is cleaned heating in an electric furnace at 1100° F for 30 . Strips are soaked in 0·1 M KH_2PO_4, dried and ed in a desiccator ready for use. A miscible ent system composed of various amounts of ethylformamide (0 to 3% v/v) in benzene is able for most steroids. Chromatography is ducted at room temp., solvents are mixed ediately before use and poured into a covered s jar of suitable size, the test soln. is spotted the lower edge, and the strip is dried and ed into the solvent, without preliminary ilibration, for ascending development. A strip m long is completely developed in approx. 15 . The solvents are evaporated, the paper is yed with conc. H_2SO_4 and heated over a hot e until the acid is volatilised. The charred oids appear as grey - black spots on a white kground. Average R_F values for a number of oids are tabulated. H. F. W. Kirkpatrick

70. **Double isotope derivative assay of aldosterone iological extracts.** B. Kliman and R. E. Peter-(Nat. Inst. of Arthritis and Metabol. Diseases, nesda, Md., U.S.A.). *J. Biol. Chem.*, 1960, **235** 1639–1648.—Dried extracts of plasma or urine acetylated with [^{3}H]acetic anhydride in the ence of pyridine to convert the aldosterone the labelled diacetate. A measured amount of sterone [^{14}C]diacetate is then added, and the bly-labelled steroid is purified by paper chroma-aphy. The sample is then treated with a 0·5% of CrO_3 in acetic acid to form a mono-acetate ation product, and this is re-chromatographed separate the product from other ^{3}H-labelled material. The contents of ^{3}H and ^{14}C are determined by counting in a liquid scintillation spectrometer. The amount of aldosterone present in the original extract is calculated from the determination of the amount of ^{14}C indicator lost during purification, the yield of ^{3}H radioactivity, and the specific activity of the [^{3}H]acetic anhydride. The assay needs 5 to 30 ml of human urine or 2 to 3 ml of dog adrenal vein plasma. It is applicable to 0·01 μg of aldosterone, and has the accuracy and precision of radio-isotopic techniques. J. N. Ashley

271. **Method of determining aldolase and a means of expressing its activity.** E. P. Korzunova (Smolensk Med. Inst.). *Lab. Delo*, 1960, **6** (3), 21–23.—Alterations are suggested to the method of Sibley and Lehninger (*J. Biol. Chem.*, 1949, **177**, 859). The soln. for incubation at 37° consists of barbitone buffer (pH 6·8) (1·5 ml), 0·05 M fructose 1,6-diphosphate (Na salt) (0·25 ml), 0·056 M hydrazine sulphate (0·25 ml), 0·002 M iodoacetic acid (0·25 ml), H_2O (0·05 ml) and test soln. (0·2 ml). The procedure is then as described by Sibley and Lehninger. A. Burwood-Smith

272. **Notes on the hydroxamate assay for amino-acid-activating enzymes.** I. D. Raacke and J. Bové (Virus Lab., Univ. of Calif., Berkeley, U.S.A.). *Experientia*, 1960, **16** (5), 195–196 (in English).—Sources of error in the hydroxamate assay are pointed out. The final pH (0·9) of the reaction mixture for the determination of hydroxamate after the addition of $FeCl_3$ is important. Over the pH range 0·9 to 0·3 there is a 20% decrease in colour yield per 0·1 pH unit. The apparent activity is also reduced to one-third when hydroxy-ammonium chloride soln. that has been stored for 21 days is used for the assay. In addition, the tissue extracts may destroy the amino-acid hydrox-amates formed. P. Nicholls

273. **Rapid method for the quantitative determination of serum alkaline phosphatase.** B. Klein, P. A. Read and A. L. Babson (Vet. Admin. Hosp., Bronx, N.Y., U.S.A.). *Clin. Chem.*, 1960, **6** (3), 269–275.—A commercial tablet form (Phosphatabs) of buffered phenolphthalein phosphate is used, the amount of phenolphthalein liberated under controlled conditions being determined photometrically to give a measure of the enzyme activity. H. F. W. Kirkpatrick

274. **Colorimetric determination of serum isocitric dehydrogenase.** T. H. Taylor and M. E. Friedman (Dr. Milton J. Goodfriend Res. Lab., Lebanon Hosp., New York, U.S.A.). *Clin. Chem.*, 1960, **6** (3), 208–215.—A method is described in which the α-oxoglutaric acid formed during the reaction is determined with 2,4-dinitrophenylhydrazine. The results are related directly to the spectrophotometric units of Wolfson and Williams-Ashman (*Proc. Soc. Exp. Biol., Med.*, 1957, **96**, 231). H. F. W. Kirkpatrick

275. **Colorimetric assay of coenzyme I and II dependent enzymes.** J. King (North Lonsdale Hospital, Barrow-in-Furness, England). *J. Med. Lab. Technol.*, 1960, **17** (2), 89–92.—Coenzyme I (diphosphopyridine nucleotide) and coenzyme II (triphosphopyridine nucleotide) form condensation products with 2,4-dinitrophenylhydrazine, and the effect of this on the colorimetric determination with 2,4-dinitrophenylhydrazine of enzymes requiring

these coenzymes is discussed. A modified standard curve for the determination of lactic dehydrogenase by the method of King (*Anal. Abstr.*, 1960, **7**, 3416) is suggested. R. A. BRENAN

276. **Quantitative determination of coenzyme A by sorbyl coenzyme-A formation.** S. J. Wakil and G. Hübscher (Inst. for Enzyme Res., Univ. of Wisconsin, Madison, U.S.A.). *J. Biol. Chem.*, 1960, **235** (6), 1554–1558.—The spectrophotometric assay depends on the formation of sorbyl coenzyme A by interaction of coenzyme A with sorbic acid in the presence of the fatty acid-activating enzyme, adenosine triphosphate and Mg^{2+}, and measurement of the sorbyl coenzyme A at 300 mμ. The method is applicable to 1 to 15 mμmoles of coenzyme A, and can be used with tissue extracts. J. N. ASHLEY

See also Abstracts—**13**, Inorg. cations in biological fluids. **67**, Scintillation counting of ^{14}C. **170**, Detection of phosphate esters. **179**, I.r. spectra of fungal pigments. **333**, Determination of vitamin B_{12}. **352**, Fractionation of nucleic acids. **365**, Zaffaroni-type solvent system for paper chromatography. **370**, Use of bromophenol blue for spot tests. **385**, Tank for electrophoresis of proteins. **386**, Photometry of lipid electropherograms. **393**, Alkaline-earth metals in biological material.

Pharmaceutical analysis

277. **Flame-photometric examination of pharmaceutical materials.** E. Pungor and E. Király (Inst. f. anorg. u. anal. Chem., L. Eötvös Univ., Budapest, Hungary). *Arch. Pharm., Berlin*, 1960, **293** (5), 494–501.—The effects of acetic and ascorbic acids, H_3BO_3, $KMnO_4$ and Ca lactate in various concn. on the mol. and atomic emissions of Na^+ (and in some cases of Ca^{2+} and K^+) are examined. Emissions of NaCl are not affected by the presence of ascorbic acid. Suitable methods are given for the elimination of the various forms of interference caused by the other compounds mentioned. A suitable formula is given for dealing with the interference with the emissions of NaCl caused by the presence of KOH. The presence of >30% of ethanol causes undue interference with the emisssion of standard and test soln. The Zeiss flame-photometer gives satisfactory results with the direct use of an injector-atomiser, without a cloud-chamber atomiser. P. S. ARUP

278. **Radiochromatographic evaluation of alkaloids in tincture and extract of nux vomica.** M. Šaršúnová, J. Tölgyessy and J. Majer (Inst. Anal. Chem., Pharm. Fac., Bratislava, Czechoslovakia). *Pharm. Acta Helv.*, 1960, **35** (6), 271–275 (in German).—Apply the tincture or the diluted extract to Whatman No. 1 paper, dry the spot in warm air and develop with water-saturated n-butanol by the descending technique for 16 hr. Immerse the chromatogram for 3 min. in a soln. of molybdophosphoric acid labelled with ^{32}P, remove the excess of reagent by washing with 2·5% HCl, allow to dry and scan with a Geiger - Müller counter. Determine the concn. of brucine and strychnine in the sample from the counts of radioactivity at the corresponding maxima. The recovery is about 95% with an error of $\simeq \pm 3\%$. A. R. ROGERS

279. **The use of paper chromatography in studying the stability of apomorphine.** F. Machovičová, O. Mohelská and V. Parrák (State Inst. Control of Drugs, Bratislava, Czechoslovakia). *Československ. Farm.*, 1960, **9** (5), 243–246.—Apomorphine (**I**) can be separated from an oxidation product (**II**) formed in unstabilised aq. soln. by paper chromatography of the test soln. (containing 100 to 200 μg of **I**) with n-butanol - formic acid - water (77 : 13 : 10) (R_F values 0·5 and 0·7 for **I** and **II**, respectively) or n-butanol - acetic acid - water (4 : 1 : 5) (R_F values 0·65 and 0·85). Dragendorff's reagent gives an orange spot with **I** and a green one with **II**. For injection soln. stabilised with ascorbic acid or mannitol, the best spray reagent is bromophenol blue soln. containing borate, which gives yellow spots with ascorbic acid and mannitol, and a green one with **I** on a blue background. J. ZÝKA

280. **Colorimetric method for the determination of atropine by means of Reinecke's salt.** S. Bartkowicz (Dept. of Appl. Pharm., Med. Acad., Cracow). *Dissert. Pharm.*, 1960, **12** (2), 113–120.—Ammonium reineckate (**I**) is used for the determination of atropine in 1% H_2SO_4. **I** soln. (0·5%) is added to the test soln., the mixture is placed in a refrigerator for 30 min., and the ppt. is collected on a glass filter, washed with cold water and dissolved in acetone. The extinction is then measured against a reagent blank. Alternatively, the extinction of the **I** soln. is measured before and after pptn. by the test soln. The difference is proportional to the amount of atropine reineckate pptd. B. K.

281. **Spectrophotometric determination of ephedrine hydrochloride.** R. C. Shah, P. V. Raman and S. B. Gandhi (Mac Laboratories Private Ltd., Bombay). *Indian J. Pharm.*, 1960, **22** (6), 140–141.—The extinction at 207 mμ of a soln. in 0·1 N HCl is measured in a Beckman spectrophotometer model G 2400, with DU photomultiplier combination G 4700, with a hydrogen discharge tube as a light source and 1-cm silica cells. Beer's law is obeyed in the range 5 to 20 μg per ml. Pseudoephedrine shows exactly the same u.v. absorption as ephedrine. The procedure has been successfully applied to the determination of ephedrine hydrochloride in tablets, elixir, ointment and nasal drops. A. R. ROGERS

282. **Colorimetric determination of dihydroxypropyltheophylline [diprophylline] by means of molybdophosphoric acid.** T. Pelczar (Dept. of Inorg. and Anal. Chem., Med. Acad., Kraków). *Dissert. Pharm.*, 1960, **12** (2), 107–111.—Diprophylline (**I**) ($\simeq$10 mg) is dissolved in water, conc. HCl is added, the soln. is heated and molybdophosphoric acid is added. The cooled ppt. is washed with dil. HCl and dissolved in 0·1 N NaOH. A soln. of ammonium molybdate in H_2SO_4 is added, followed by a soln. of ascorbic acid, then the mixture is boiled for 1 min., cooled, and measured colorimetrically. This method was used for the determination of **I** in tablets and injections. B. K.

283. **Reactions of deoxy-sugars. Application to the determination of glycosides.** H. Wachsmuth and L. van Koeckhoven (Lab. de Chim. et de Biochim., Hôpital Stuivenberg, Anvers). *J. Pharm. Belg.*, 1959, **14** (3–4), 165–172.—Many reagents are studied for the characterisation and identification of deoxy-sugars, glycosides and similar substances. These include mineral salts, aldehydes, indole derivatives, heteropoly-acids, and the chlorides of

Bi and As. Colour reactions and test procedures described. Quantitative methods have been posed for two of the reagents, *viz*, sodium molybe and phthalaldehyde. In each case the extinc- is compared with that of a standard sample. *cedure with sodium molybdate*—Dissolve the xy-sugar or glycoside (25 to 200 μg) in 2 ml of ic acid. Add 0·5 ml of molybdate soln. (0·5 ml 50% aq. sodium molybdate in 25 ml of acetic l), and then 0·5 ml of acetic acid containing of H_2SO_4. Set aside for 3 hr., make up to l with acetic acid and measure the extinction at mμ in a 1-cm cell for digitoxose (**I**), digitoxin or digoxin. *Procedure with phthalaldehyde*: (*a*) **I** *and* **II**—Dissolve the sample (25 to 200 μg) cetone and evaporate to dryness. Add 0·1 ml reagent (100 mg of phthalaldehyde in 4 ml of nol) and 6 ml of HCl. Set aside for 90 min. read the extinction at 525 mμ for **II**, or 530 for **I**. (*b*) *For* **II**—This procedure is used when hod (*a*) gives a cloudy soln. Add 0·2 ml of ent (0·2 g of phthalaldehyde in 4 ml of ethanol) 6 ml of HCl to the sample (25 to 200 μg). aside for 1 hr., add 4 ml of butanol, set aside 20 min., and read the extinction at 520 mμ. hod (*b*) may be slightly modified for application eoxyribose. R. A. Hendey

34. **Automated system for continuous determination of penicillin in fermentation media using coxylamine reagent.** A. O. Niedermayer, F. M. so-Alesi, C. A. Lendzian and J. M. Kelly (Squibb . for Med. Res., New Brunswick, N.J., U.S.A.). *l. Chem.*, 1960, **32** (6), 664–666.—The use of a nicon AutoAnalyser is described, in which the plex obtained by the reaction of penicillin (**I**) hydroxylamine and Fe^{3+} (*cf.* Boxer and Everett, . *Abstr. C*, 1949, 404) is measured at 480 mμ. 's law is obeyed over the range 250 to 10,000 s of **I** per ml. The method, which is preferred ne based on differences in the iodine uptake of ve and degraded **I** (*cf.* Ferrari *et al.*, *Anal. Abstr.*, **7**, 2431), has been used extensively for the y of fermentation media. Results are in factory agreement with those obtained microogically. The coeff. of variation is 2·5%. A. R. Rogers

35. **Determination of chloramphenicol in various dicaments.** M. Andrey and A. Mirimanoff . de Pharm. Galén., Univ. de Genève, Switzer-). *Pharm. Acta Helv.*, 1960, **35** (6), 276–283 French).—Extract suppositories with benzene use the benzene-insoluble portion; extract the ents of capsules with absolute ethanol and orate to dryness. Ignite the dried residue with CO_3, extract with H_2O and determine Cl^- by method of Volhard. The precision of the od is within $\simeq \pm 6\%$. A. R. Rogers

36. **Rifomycin. VII. Spectrophotometric analysis of rifomycin B.** G. G. Gallo, P. Sensi and P. aelli (Lab. Ric., Lepetit S.P.A., Milano, Italy). *naco, Ed. Prat.*, 1960, **15** (5), 283–291.—trophotometry is applicable to the determination of rifomycin B as pure substance, and in maceutical preparations, fermentation broths biological fluids. In pure solution in phose buffer at pH 7·3 (2·28 g of $NaH_2PO_4.H_2O$ and g of $Na_2HPO_4.12H_2O$ per litre), $E^{1\%}_{1\,cm}$ at 425 mμ 220. Pharmaceutical preparations are dispersed he buffer and the rifomycin is extracted into l acetate after acidification to pH 2. For mycin B in ethyl acetate, $E^{1\%}_{1\,cm}$ at 414 mμ = 145. If the ethyl acetate extract is coloured (*e.g.*, in the assay of fermentation broths and biological fluids) the rifomycin is re-extracted into pH 7·3 buffer for spectrophotometric determination. The absorption spectra of rifomycin B in various solvents and at different pH values are reproduced over the range from 250 to 500 mμ.

VIII. Fluorimetric assay of rifomycin B. P. Sensi, G. Coronelli and A. Binaghi. *Ibid.*, 1960, **15** (5), 292–300.—The method is based on measurement of the fluorescence of the triacetyl derivative in ethyl acetate soln., which has an emission maximum at 500 mμ. Solid samples are dissolved in 0·067 M phosphate buffer at pH 7·3 (approx. 20 μg of rifomycin B per ml). The soln. is acidified to pH 2 with 10% HCl and extracted twice with an equal volume of ethyl acetate. The extract is dried with anhyd. Na_2SO_4, filtered and made up to a known vol. An aliquot containing from 50 to 500 μg of rifomycin is measured into a 50-ml flask, acetic anhydride (0·1 ml) and pyridine (0·2 ml) are added and the mixture is diluted to vol. with ethyl acetate and set aside for 2 hr. The fluorescence at 500 mμ is then measured in a spectrofluorimeter and compared with the fluorescence of standards treated similarly. The graph of fluorescence *vs.* concn. is linear over the range from 1 to 10 μg per ml. The standard deviation at the 10-μg level is 0·42 μg per ml. E. C. Apling

287. **Methods of insulin assay.** G. A. Stewart (Biological Control Laboratories, The Wellcome Foundation, London). *Brit. Med. Bull.*, 1960, **16** (3), 196–201.—A review, with 83 references. D. E. Evans

288. **Determination of some alkaline organic salts in non-aqueous medium.** R. Vasiliev, E. Şişman, M. Jecu and I. Chialda (Inst. Pharm. Res., Bucharest). *Rev. Chim., Bucharest*, 1960, **11** (6), 347.—To approx. 1·5 to 2 milli-equiv. of organic salt dissolved in 20 ml of ethanol is added $\simeq$2 g of phenol and the mixture is titrated with 0·1 N $HClO_4$ in dioxan to thymol blue. Errors of $\simeq \pm 0{\cdot}13\%$ are shown for a number of salts. H. Sher

289. **Paper chromatography of derivatives of barbituric acid.** A. Macek (Forschungsinst. f. Pharm. u. Biochem., Prague, Czechoslovakia). *Arch. Pharm., Berlin*, 1960, **293** (5), 545–550.—R_F values obtained with the use of benzene, $CHCl_3$, or benzene - $CHCl_3$ (1 : 1) as descending solvents on paper impregnated with formamide are given for 41 substitution derivatives of the acid. The values for di- and tri-substitution derivatives are spread over wider ranges than those obtained by the usual techniques. Barbituric acid and its mono-substitution derivatives remain at the starting point, and can subsequently be treated with other solvents. Constants are given for 20 possible substituting groups, showing their influence on the R_F value (obtained with $CHCl_3$ as solvent) when replacing an ethyl group in the C_5 position. P. S. Arup

290. **The application of alternating-current polarography in the determination of phenobarbitone in dosage forms.** N. G. Lordi, E. M. Cohen and B. L. Taylor (College of Pharmacy, Rutgers, The State Univ., Newark, N.J.). *J. Amer. Pharm. Ass., Sci. Ed.*, 1960, **49** (6), 371–375.—Prepare a soln. containing phenobarbitone (0·2 to 0·4 mg) in a M soln. of KNO_3 in borate buffer of pH 7·9 (25 ml) and submit it to polarography with an applied alternating current of 0·03 V r.m.s. and a rate of

scan of 0·00062 V per sec. Calculate the concn. of phenobarbitone from the peak current by reference to a standard curve. The accuracy is $\simeq \pm 2\%$. Caffeine, diphenylhydantoin, aminophylline, theobromine sodium salicylate, thiamine, polyoxyethylene glycol 400 and halides interfere.

A. R. Rogers

291. **Determination of amidopyrine in non-aqueous medium.** R. Vasiliev, V. Scintee and I. Chialda (Inst. Pharm. Res., Bucharest). *Rev. Chim., Bucharest*, 1960, **11** (6), 347.—*Procedure*—Dissolve between 0·3 and 0·4 g in 20 ml of $CHCl_3$ and titrate with 0·1 N $HClO_4$ in dioxan until methyl red changes to red - violet. The error is $\pm 0{\cdot}04\%$. It is claimed that the method can be used for mixtures containing salts of alkaloids, caffeine and other purine and barbituric acid derivatives.

H. Sher

292. **Colorimetric determination of 4-aminophenazone and 4-formamidophenazone in amidopyrine.** J. Gasparič (Res. Inst. Org. Synth., Pardubice-Rybitví, Czechoslovakia). *Českosl. Farm.*, 1960, **9** (5), 241–243.—*Procedure*—Dissolve amidopyrine (4 g) in 0·4 N HCl (50 ml). Cool an aliquot (10 ml) to 0° to 5° and add $NaNO_2$ soln. (2%) (2 ml). After 5 min. add a soln. of phenyl-J-acid (dissolve 6 g of anhyd. Na_2CO_3 in 80 ml of H_2O, cool with ice, add 100 mg of phenyl-J-acid and dilute with H_2O to 100 ml; the soln. must be freshly prepared and stored at 0° to 5°) (10 ml), dilute with H_2O to 50 ml and after 10 to 15 min. measure the extinction at 490 mμ for 4-aminophenazone. Heat another aliquot for 30 min. on a boiling-water bath to hydrolyse 4-formamidophenazone to 4-aminophenazone, cool and proceed as described above to determine the sum of the two impurities. Beer's law is obeyed over the range 0 to 200 μg of 4-aminophenazone in 50 ml. The max. error is $\pm 5\%$.

J. Zýka

293. **Determination of some tranquillisers by ammonia distillation.** P. M. Parikh, G. T. Rajkotia and S. P. Mukherji (Zandu Pharmaceutical Works Ltd., Bombay). *Indian J. Pharm.*, 1960, **22** (6), 148–149.—Heat the sample (250 mg of meprobamate or 400 mg of mephenesin carbamate or methocarbamol) under reflux with 50% (v/v) H_2SO_4 soln. (10 ml) for 10 min., dilute with H_2O (40 ml), add 50% (w/v) NaOH soln. (25 ml), distil the liberated NH_3 into 0·1 N H_2SO_4 (50 ml) and titrate the excess of acid with 0·1 N NaOH, with methyl red as indicator. Perform a blank determination. The results are comparable with those obtained by the Kjeldahl method and are produced more quickly.

A. R. Rogers

294. **Photometric determination of Novazin (2-diethylaminoethyl phenothiazine-10-carboxylate).** M. Sterescu, M. Popa and H. Aftalion. *Rev. Chim., Bucharest*, 1960, **11** (6), 346–347.—The method is based on pptn. with ammonium reineckate. The ppt. is dissolved in acetone and the extinction is measured at 530 mμ with water as blank. The error is $\pm 3\%$.

H. Sher

295. **pH-Induced shifts in ultra-violet maxima for determination of isoniazid and acetylisoniazid in mixtures.** Q. C. Belles and M. L. Littleman (Leahi Hosp., Honolulu, Hawaii, U.S.A.). *Anal. Chem.*, 1960, **32** (6), 720–721.—Acetylisoniazid (**I**) and isoniazid (**II**) exhibit maxima at 265 mμ in acid soln. and at $\simeq 300$ mμ in alkaline soln. The compounds make this shift to 300 mμ at different pH values, and can thus be differentiated; **I** makes a complete shift in max. at pH 10 while **II** remains unaffected; the complete shift for **II** occurs at pH 12. These u.v. differences were used to determine the compounds in blood, serum or urine. The method is useful when the sensitivity required is < 1 μg per ml.

G. P. Cook

296. **Photometric determination of hydrochlorothiazide and its hydrolysis product.** C. R. Rehm and J. B. Smith (Ciba Pharmaceutical Products, Inc., Summit, N.J.). *J. Amer. Pharm. Ass., Sci. Ed.*, 1960, **49** (6), 386–389.—The u.v. absorption max. at 271 mμ in methanol or H_2O may be used for the quant. determination of hydrochlorothiazide (**I**) in the absence of the main hydrolysis product 4-amino-6-chlorobenzene-1,3-disulphonamide (**II**); Beer's law is obeyed over the range 1 to 10 mg per litre. To determine **II** in the presence of **I**, dissolve the sample in methanol or 2-methoxyethanol and dilute with H_2O; mix the soln. (2 to 5 ml containing $\simeq 0{\cdot}1$ mg of **II**) with 1% $NaNO_2$ soln. (1 ml) and N HCl (5 ml), set aside for 3 to 5 min., add 2% ammonium sulphamate soln. (1 ml), mix, set aside for 3 min., add a fresh 0·5% soln. of chromotropic acid (disodium salt) (1 ml) and N Na acetate (10 ml), dilute to 50 ml with H_2O, mix, and measure the extinction at 500 mμ against a reagent blank. Treat similarly a standard soln. of **II**. To determine **I** and **II** together, mix a soln. of the sample ($\simeq 0{\cdot}2$ mg) in 0·5 N aq. NH_3 (2 ml) with 0·7% hydroxylammonium chloride soln. (0·5 ml), heat at 100° for 60 min. in a closed flask, then cool, and determine **II** as described above. Calculate the amount of **I** by difference. The precision is $\simeq \pm 1\%$.

A. R. Rogers

297. **Identification and spectrophotometric determination of hydrochlorothiazide (Hydrodiuril).** M. Sterescu, V. Topa, S. Arizan, S. Iacobescu and Z. Teodorescu (Inst. Phys., Bucharest). *Rev. Chim. Bucharest*, 1960, **11** (5), 296–297.—The proposed method is based on the characteristics of the absorption curves of a soln. of hydrochlorothiazide (**I**) in 0·0002 N NaOH, *viz*, max. extinctions E_1 at 222 mμ and E_2 at 274 mμ and the relationship $1{\cdot}9 < E_1/E_2 > 2$. *Procedure*—The sample (0·25 g) is dissolved in 50 ml of 0·1 N NaOH; 2 ml is then diluted to 100 ml with water and 5 ml of this soln. is then further diluted with 50 ml of water (at concn. > 10 μg per ml, **I** is not completely soluble). The pure compound gives values for $E^{1\%}_{1cm}$ of $E_1 = 0{\cdot}99$ and $E_2 = 0{\cdot}495$. The concn. of a sample in μg per ml is then given by $10/0{\cdot}99\ E_1$ or $10/0{\cdot}495\ E_2$. The results obtained with a Zeiss spectrophotometer gave errors of $< 2\%$.

H. Sher

298. **New method for the determination of 4-amino-6-chlorobenzene-1,3-disulphonamide.** S. Arizan and M. Sterescu. *Rev. Chim., Bucharest*, 1960, **11** (5), 297–298.—The method for the determination of 4-amino-6-chlorobenzene-1,3-disulphonamide (**I**) as an impurity in hydrochlorothiazide is based on the colour reaction given by **I** when diazotised and coupled with *N*-1-naphthylethylenediamine (**II**). *Preparation of a standard curve*—**I** (0·025 g) is dissolved in 5 ml of N NaOH and diluted to 250 ml, from which aliquots of 0·1, 0·2 and 0·3 ml are taken. Each is diluted to 2 ml with 0·02 N NaOH, stirred and treated with 2 ml of 0·1 N HCl and 0·4 ml of a 1% soln. of $NaNO_2$, stirred and set aside for 5 min. Each is treated with

nl of a 0·4% sulphamic acid soln., stirred for 1 , and 0·4 ml of a 0·1% aq. soln. of the di-hydro- ride of **II** is added, with stirring. The extinc- s of the rose-coloured soln. are read after 10 at 530 mμ in a 1-cm cell against water. *Deter- ination of* **I** *in chlorothiazide*—The sample (0·2 g) ssolved in 2 ml of N NaOH and diluted to 100 ml. soln. (1 ml) is treated with 1 ml of 0·02 N NaOH the procedure for the standard curve is followed. error is $\gtrsim \pm 4\%$. H. Sher

99. **Microchemical reactions for some new oral etics.** H. Groenewegen (Apoth. Ziekenf. "De iarding," Den Haag, Netherlands). *Pharm. kbl.*, 1960, **95** (11), 345–350.—Microchemical tions are described for chlorothiazide (**I**), rochlorothiazide (**II**), hydroflumethiazide (**III**) acetazolamide (**IV**). The following reagents are l—aq. NH_3 (for **I**, **III** and **IV**), Cu acetate and NH_3 (for **I**), Zn acetate and aq. NH_3 (for **I**, and **IV**), $AgNO_3$ and aq. NH_3 (for **I**), ethan- iine and $AgNO_3$ (for **I** and **IV**), ethylenediamine $AgNO_3$ (for **I**, **III** and **IV**) and the reagents of chardat and Dragendorff - Amelink (for **I** and Twenty illustrations showing the crystal form he products are given. M. J. Maurice

00. **Volumetric determination of nitrofurazone.** Spacu and G. Teodorescu (Univ. "C.I. Parhon," harest, Romania). *Z. anal. Chem.*, 1960, **174** 321–322 (in German).—Nitrofurazone (5-nitro- rfuraldehyde semicarbazone) (**I**) is determined hydrolysing to liberate an equivalent of hydr- e, which is titrated with standard chloramine-T . *Procedure*—To the sample soln. containing to 0·07 g of **I** in dil. acetic acid (20 to 50 ml) conc. HCl (20 to 30 ml) and water (20 to 30 ml). t to 70° to 80°, add methyl red - methylene indicator, and titrate with 0·1 N chloramine T, ing more indicator just before the end-point; l of 0·1 N chloramine T ≡ 0·004953 g of **I**. maximum error for the given range is −0·4%. W. T. Carter

01. **Quantitative colorimetric determination of dual 9α-fluoroprednisolone and 9α-fluorohydro- isone in triamcinolone samples.** C. J. Sih, S. C. and R. E. Bennett (Squibb Inst. for Med. , New Brunswick, N.J., U.S.A.). *Anal. Chem.*, 0, **32** (6), 669–671.—Dissolve the sample, con- ing 10 to 100 μg of 9α-fluoroprednisolone (**I**) or uorohydrocortisone (**II**) or both, in methanol ml), cool in ice and add successively H_2O ml), 0·01% fructose soln. (0·5 ml) and H_2SO_4 6 vol. of conc. H_2SO_4 with 1 vol. of H_2O) ml). Cool in ice for 10 min., with occasional ring, set aside at room temp. for ≃10 min. and heat at 60° for exactly 5 min. Cool in water, 3% L-cysteine hydrochloride soln. (0·1 ml) and aside overnight at room temp. Dilute with (1·2 ml) and measure the extinction at 548 mμ at 625 mμ. Simultaneously run a blank con- ing pure triamcinolone (**III**). The absorption 25 mμ is due to **I** and that at 548 mμ is due to us **II**. When the amount of **III** present is >50 s the amount of **I** plus **II**, the chromogens do obey Beer's law, and a standard curve must be pared. The precision is ≃±3%. Many other oids give colour reactions with the reagents. A. R. Rogers

02. **Analysis of anaesthetic mixtures by gas matography.** E. R. Adlard and D. W. Hill ell" Research Ltd., Thornton Research Centre, Chester, England). *Nature*, 1960, **186**, 1045.—To separate O and N, use a column (1 ft. × 0·25 in.) of 52 to 60-mesh Linde 5A molecular sieve at room temp. To separate CO_2, N_2O and O, use a column (20 ft. × 0·25 in.) containing 20% of dimethyl sulphoxide on 52 to 60-mesh "SIL-O-CEL" at 20°. When ethyl ether and fluothane are also present, use a column (2 ft. × 0·25 in.) containing 15% of dinonyl phthalate on firebrick at 75° in parallel with the dimethyl sulphoxide column. With H as carrier gas at a flow rate of 30 ml per min., analysis is complete in 4·5 min. A. R. Rogers

303. **Application of paper electrophoresis to the detection of heavy metals as impurities in pharmaceutical preparations.** D. Małysz (Drug Res. Inst., Warsaw). *Acta Polon. Pharm.*, 1960, **17** (3), 221–228.—Conditions for the use of electrophoresis are investigated. The best results are given by the following procedure. Incinerate 1 g of sample, add a few drops of conc. HCl, dry, dissolve the ash in 1 ml of 0·1 N HCl, with heating, and filter through a small filter. Transfer 0·05 to 0·2 ml of the filtrate to Whatman No. 1 paper and apply a potential of 300 V for 3 hr.; use Na citrate buffer (10 g of citric acid and 3·5 g of NaOH in 1 litre of water). The following reagents were used for detection (figures in parentheses show the smallest detectable amount)—*p*-phenetidine for Fe (0·5 μg), Cu (0·25 μg) and Zn (0·25 μg); 8-hydroxyquinoline for Fe (0·25 μg), Cu (0·5 μg) and Zn (0·25 μg); Na diethyldithiocarbamate for Fe (0·25 μg) and Cu (0·1 μg); rhodizonic acid for Pb (0·2 μg); and dithizone for Pb (0·25 μg) and Zn (0·1 μg). A modification is described for preparations containing Na and Ca in which the heavy metals are pptd. with H_2S in the presence of mercury salts. B. K.

304. **Complexometric method for the assay of strong solution of lead subacetate B.P.** C. R. Mehta, N. V. Mankad and M. B. Devani (Dept. of Pharm. Chem., L. M. Coll. of Pharmacy, Ahmedabad, India). *Indian J. Pharm.*, 1960, **22** (5), 123–124.—*Procedure*—Dilute the sample (1 g) with cation-free H_2O (20 ml) and titrate with 0·1 M EDTA (disodium salt), with continuous shaking, until the turbidity changes to a ppt. with a clear supernatant liquid (*a* ml). Continue the titration with vigorous shaking until the ppt. dissolves and a clear soln. is obtained (*b* ml). The results for total lead as Pb (calculated from *b*) are 0·44% (absolute) higher than by the B.P. method, but agree closely with those by a gravimetric sulphate method. The results for alkalinity as PbO, calculated from $(b - a)$, are also 0·44% higher than by the B.P. method. The standard deviations, based on 10 replicates, were 0·024% for the proposed method and 0·033% for the B.P. method. A. R. Rogers

305. **Analysis by gas chromatography of lozenges "a l'éther."** L. Maricq and L. Molle (Univ. Libre de Bruxelles). *J. Pharm. Belg.*, 1959, **14** (3–4), 156–158.—The chloroform, ethanol and ethyl ether contents are determined directly. *Procedure*—Introduce a lozenge into a stoppered flask containing 10 ml of H_2O. Set the flask aside for 1 to 2 hr., shaking at intervals until the lozenge has disintegrated. Centrifuge for 10 min. in a stoppered tube at 2500 to 3000 r.p.m. Inject 10 μl of the supernatant liquid into the gas stream of the chromatograph. The column (4 metres) is packed with 20% of Carbowax 1500 on Celite. The operating temp.

is 106° ± 0·5° and the carrier gas is H, at a flow rate of 25 ml per min. The content of $CHCl_3$ is obtained by comparing the peaks with a standard curve obtained in the same way; the amounts of ethanol and ether can also be measured. Results compare well with those found by a preliminary distillation followed by gas chromatography.

R. A. Hendey

306. **Estimation of the biological potency of isomerised vitamin-A palmitate, in aqueous multivitamin dispersions, from maleic values.** S. R. Ames, W. J. Swanson and R. W. Lehman (Distillation Products Industries, Div. of Eastman Kodak Co., Rochester, N.Y.). *J. Amer. Pharm. Ass., Sci. Ed.*, 1960, **49** (6), 366–368.—The relative biopotency (y) is the potency determined by the slope-ratio liver-storage method (*cf.* Ames and Harris, *Anal. Abstr.*, 1956, **3**, 3202) expressed as a percentage of the chemical potency determined by the blue colour with $SbCl_3$. The maleic value (x) is determined by the method described by Embree *et al.* ("Methods of Biochemical Analysis," Vol. 4, Interscience Publ. Inc., New York, 1957, p. 43). Studies of stored multivitamin dispersions showed that y and x are related by the cubic regression equation—

$$y = 99{\cdot}5 - 0{\cdot}2x - 0{\cdot}051x^2 + 0{\cdot}000768x^3.$$

The isomerisation of all-*trans* vitamin-A palmitate in aq. dispersions appears to reach an equilibrium; y does not fall below 60% even after prolonged storage.

A. R. Rogers

307. **Determination of nordihydroguaiaretic acid antioxidant in oily vitamin preparations.** A. Engelund (Denmark Apothekerforenings Kontrollab., Copenhagen). *Pharm. Acta Helv.*, 1960, **35** (6), 284–291 (in English).—Extract nordihydroguaiaretic acid (**I**) from fatty material with hot 63% ethanol, cool and filter the extract, and apply an aliquot (3 to 6 μl containing ≃2 μg of **I**) to S. & S. paper No. 2043B. Develop with a 2·5% aq. soln. of ethyl acetoacetate by the descending technique for 2 hr., dry the chromatogram, spray with ethanolic $AgNO_3$ soln., dry again and spray with aq. NH_3. Measure the area of the light-brown spot at R_F ≃0·3; the area is directly proportional to the log of the amount of **I**. The error is ≃±15%.

A. R. Rogers

See also Abstracts—**153**, Mercury in org. compounds. **157**, Ethanol in aq. media. **176**, Polycyclic hydrocarbons in tobacco smoke. **185**, I.r. spectra of naphthols. **200**, Ethanol in volatile oils. **236**, Identification of corticosterone sulphate. **268**, Chromatography of medicinal steroids. **331**, Determination of thiamine. **333**, Determination of vitamin B_{12}. **342**, Determination of warfarin.

Food

Foods and food additives, beverages, edible oils and fats, vitamins.

308. **Studies on legume starches. IV. Separation and determination of amylose and amylopectin. (2). Iodine coloration and potentiometric titration.** Sin'itiro Kawamura and Minoru Tada (Fac. of Agric., Kagawa Univ., Miki-cho, Kagawa-ken). *J. Agric. Chem. Soc. Japan*, 1959, **33** (4), 296–300.—Amylose and amylopectin were separated from eight kinds of legume starch and potato starch by dispersion in 1% NaOH soln. and pptn. with butanol. The amounts of starch, amylose and amylopectin were determined by colorimetry (McCready *et al.*, *Anal. Chem.*, 1950, **22**, 1156) and potentiometric titration (Bates *et al.*, *J. Amer. Chem. Soc.*, 1943, **65**, 142).

S. Natori

309. **Analytical applications of the complexans. XXVII. Determination of heavy metals in samples of cane and beet syrup with ADCT [1,2-diaminocyclohexane-*NNN'N'*-tetra-acetic acid] and AEGT [1,2-di-(2-aminoethoxy)ethane-*NNN'N'*-tetra-acetic acid].** F. Bermejo Martínez, M. Paz Castro and R. Rey Mendoza (Fac. de Ciencas, Santiago de Compostela, Spain). *Inf. Quim. Anal.*, 1960, **14** (3), 61–63, 80.—The syrup (0·5 g) is oxidised with HNO_3 and H_2SO_4 and evaporated to dryness, and the residue is dissolved in dil. HCl and diluted to 250 ml. To aliquots (25 ml) are added 0·01 M ADCT (disodium salt) (5 ml) and 0·01 M AEGT (disodium salt) (5 ml); the pH is adjusted to 11 to 12 by addition of aq. NH_3 and murexide is added as indicator. The excess of complexan is determined by back-titration with 0·01 M Ca^{2+}. The ADCT titration gives the sum of Al, Fe, Cu and Zn, and the AEGT titration gives the sum of Cu and Zn; Al and Fe are obtained by difference.

G. H. Foxley

310. **Examination of the occurrence of honeydew in honey.** K. C. Kirkwood, T. J. Mitchell and D. Smith (Dept. of Chem. Technol., Royal Coll. of Science and Technology, Glasgow, Gt. Britain). *Analyst*, 1960, **85**, 412–416.—The insect excretion, honeydew, collected by bees during shortage of floral nectar, affects the quality of the honey and the health of the bees. It has a dark colour and rank flavour, and can be distinguished from floral honey by its high content of ash, dextrin, sucrose and reducing sugars, its higher pH and free acidity and its dextro-rotation. The relative values of these properties for detecting honeydew in honey were examined by methods briefly described, but, owing to overlapping of the ranges, these results proved of little value for distinguishing floral honey from honeydew honey with certainty. They were therefore subjected to statistical discriminatory analysis, and mathematical investigation showed that an efficient discriminant function is given by the equation $X = -8{\cdot}3x_1 - 12{\cdot}3x_2 + 1{\cdot}4x_3$, in which x_1 is the pH, x_2 the ash (%) and x_3 the reducing sugars (%). In practice, a honey having a value of $X > 73{\cdot}1$ may be classed as floral and one having a value of $X < 73{\cdot}1$ as a honeydew honey. With samples having a value of X near 73·1 the dark colour, unpleasant flavour, dextro-rotation, high dextrin content and high acidity provide confirmatory evidence of the presence of honeydew.

A. O. Jones

311. **Correlation of anticholinesterase effect with solanine content of potatoes.** D. C. Abbott, K. Field and E. I. Johnson (D.S.I.R., Lab. of the Gov. Chemist, Clement's Inn Passage, Strand, London). *Analyst*, 1960, **85**, 375–376.—Results obtained for the solanine content of new potatoes determined by the spectrophotometric method of Baker *et al.* (*Anal. Abstr.*, 1955, **2**, 3481) differed from those determined as the anticholinesterase activity of the juice expressed as the equivalent solanine content. Investigation showed that the anticholinesterase activity appears to have an approx. direct relation to the solanine content as determined by the spectrophotometric method. The methods used

not specific for solanine, and the results cannot ve or disprove the implied opinion of Pokrovsky *okhimiya*, 1956, **21**, 683) that the anticholin-rase activity is due principally to solanine, but y indicate, contrary to the findings of Orgall *et* (*Science*, 1958, **128**, 1136), that over 90% of the l anti-esterase is pptd. at pH 9·4.

A. O. Jones

12. **Rapid determination of the protein content milk. I. Colorimetric method with Amido black and Orange G.** K. Shiga, R. Shimizu and Hamada. *Bull. Nat. Inst. Agric. Sci., Japan*, G, 1959, (18), 1–6.—Milk (10 ml), diluted 1 in was mixed with 25 ml of 0·001 M Amido black (C.I. Acid Black 1), pH 2·8, and filtered after in. The filtrate was diluted to 50 vol. and the inction was measured spectrophotometrically at mμ. This value, minus that of the blank, is a asure of the amount of dye adsorbed by the tein and is related to it by the equation $y = 33x + 0{\cdot}013$, where x is the protein determined the Kjeldahl method. Deviation from the ılts obtained by the Kjeldahl method on milk h a protein content of 1·5 to 4·5% was 0·083%. ido Black 10B was superior to Orange G.

Nutr. Abstr. Rev.

13. **Determination of sugars in fruits. II. ro-determination of glucose in fruits by the oiodite method.** Kazuo Osodo, Hidejiro Kazumi, sayuki Kotaka and Hanao Shitomi (Nat. Inst. Rural Ind., Shinjo, Yamagata, Japan). *J. ric. Chem. Soc. Japan*, 1959, **33** (3), 166–170.— conditions for the determination of glucose in presence of fructose and sucrose by hypoiodite e examined. S. Natori

14. **Identification of stored-products insects by micromorphology of the exoskeleton. X. mmon fragments of mill and grain moths.** M. rris (Dept. of Health, Education and Welfare, od & Drug Admin., New Orleans, La., U.S.A.). *Ass. Off. Agric. Chem.*, 1960, **43** (2), 444–458.— racteristics of the wings, labial palpi, legs and ennae of various flour, meal and grain moths are cribed and illustrated. A. A. Eldridge

15. **Paper-chromatographic and polarographic rmination of vanillin and ethylvanillin in food-ffs. II.** H. Woggon, K. Rauscher and U. ıler (Inst. f. Ernährung, Potsdam-Rehbrücke, llin). *Nahrung*, 1960, **4** (4), 374–388.—*Procedure* he sample (5 g) is treated with abs. ethanol ml) for 30 min. at 45°. The cooled extract is anted off and a suitable aliquot (*e.g.*, 0·1 ml) pplied to Whatman 3MM paper with a micro-er pipette (illustrated). The paper is dried and eloped by the ascending technique for 15 to 16 with isobutyl alcohol - conc. aq. NH_3 (5:3); values for vanillin are 0·33 to 0·40, and for ylvanillin 0·51 to 0·58. The spots are located er u.v. light and immediately eluted with M semicarbazide hydrochloride in 0·1 N HCl, the eluates are polarographed between −0·5 −1·0 V *vs.* the N.C.E. For vanillin (5 to 15 per ml) the mean error is ±2%, and for ethyl-illin (2 to 5 μg per ml) it is ±4%; concn. < g per ml can be determined with a mean error ±7%. Results are quoted for various flavoured ars and pudding mixtures. R. E. E.

316. **Identification of carob-bean flour in dry gravy and mayonnaise mixtures.** A. T. Czaja (Botan. Inst. der Tech. Hochschule, Aachen). *Z. Lebensmitt Untersuch.*, 1960, **112** (3), 190–194.—This flour, used in the textile industry for dressings and as a tragacanth substitute in the food and pharmaceutical industry, cannot be identified by chemical means and, as the husk and the embryo are removed during manufacture, the product loses morphological characteristics important for microscopical identification. But if the cell walls are cut lengthwise the tubular cells light up strongly under polarised light, and the cross-section shows round and oval cells which light up with varying intensity.

I. Dickinson

317. **Analytical studies of dyes by the use of infra-red spectra. I. Studies on yellow dyes for food.** Michio Suzuki, Eizo Nakamura and Yuzo Nagase (Coll. of Pharm., Kashiwagi, Shinjuku-ku, Tokyo). *J. Pharm. Soc. Japan*, 1959, **79** (9), 1116–1119.—Infra-red spectroscopic analysis of the yellow dyes Naphthol yellow S (C.I. Acid Yellow 1), Yellow AB (C.I. Solvent Yellow 5), Yellow OB (C.I. Solvent Yellow 6), tartrazine, Sunset yellow FCF (C.I. Food Yellow 3), auramine and Butter Yellow was examined by the use of KBr discs, and the dyes were detected down to ≃40 μg by their key bands at 796, 992, 1109, 1008, 903, 945 and 767 and 928 cm^{-1}, respectively.

II. Studies on green dyes for food and malachite green. Michio Suzuki, Eizo Nakamura and Yuzo Nagase. *Ibid.*, 1959, **79** (9), 1209–1211.—Guinea green B (C.I. Acid Green 1) (**I**), Light green SF (C.I. Acid Green 5) (**II**), Fast green FCF (**III**) and malachite green (**IV**) (up to 40 μg each) were similarly analysed, the absorptions at 938 cm^{-1} for **I**, 1012 for **II**, 893 for **III**, 1042 for **I**, **II** and **III**, and 945 for **IV** being used as key bands. S. Natori

318. **Analytical study of the natural red food dye alkannin.** G. Dušinský and A. Szokolay (Inst. f. Hygiene, Bratislava, Czechoslovakia). *Nahrung*, 1960, **4** (4), 356–362.—Alkannin is present in the roots of *Alkanna tinctoria* L.; it is sol. in fats and org. solvents, has a melting range of 143° to 149° and is a permitted food dye in several countries. The absorption spectra (visual and u.v.), polarographic reduction and paper and adsorption chromatography of alkannin were studied. Procedures and results are given. Extinctions at 490 and 380 mμ are suitable as a criterion of purity, polarography is suitable for quant. determination, and paper chromatography or circular chromatography on a surface of Al_2O_3 is suitable for identification in the presence of other dyes. I. Dickinson

319. **Rapid spectrophotometric method for simultaneous determination of benzoic and sorbic acids in margarine and butter.** J. B. Roos and A. Versnel (Reichsuntersuchungsanst. für Molkereiprodukte und Nahrungsfette, Leiden, Netherlands). *Dtsch. LebensmittRdsch.*, 1960, **56** (5), 128–133.—A 5-g sample is warmed slightly and washed into a 100-ml flask with 30 ml of specially purified methanol; 10 ml of H_2O and 0·5 ml of 4 N H_2SO_4 are added, the mixture is warmed on a water bath to melt the fat, then shaken vigorously for 1 min., cooled to 20° and made up to vol. with methanol. The flask is placed in ice-water to solidify the fat and the soln. is filtered through a folded 15-cm S. & S. 588 paper, the first 20 ml being discarded. The extinctions at 220, 228, 233·3, 236, 252, 258, 264, 267, 272

and 277 mμ are measured in a quartz cell of thickness giving values between 0·2 and 0·8, and are calculated to a standard 1-cm thickness. For the comparison soln., 11 ml of H_2O and 0·5 ml of 4 N H_2SO_4 are made up to 100 ml with methanol. Calculation of the results is based on maxima at 228 mμ and 272 mμ for benzoic acid, 258 mμ for sorbic acid, and an isosbestic point at 233·3 mμ for the mixed acids. Readings taken on either side of the critical wavelengths allow corrections to be made for non-specific general absorptions. Small residual corrections are required for traces of fat extracted by the solvent. Recoveries within ≃50 p.p.m. are reported. C. L. HINTON

320. **Presumptive detection of preservatives in canned cured-meat products by a simple yeast-growth test.** D. A. A. Mossel, H. Zwart and A. S. de Bruin (Central Inst. for Nutrition and Food Res., T.N.O., Utrecht, The Netherlands). *Analyst*, 1960, **85**, 410–412.—The sample is homogenised at 60° with a substrate containing glucose, meat extract, tryptone and peptonised milk agar, and the pH is adjusted to 7·0 ± 0·2 with sterile NaOH soln. A similar portion of the sample is homogenised similarly, but the pH is adjusted to 3·6 ± 0·1 with tartaric acid soln. The mixtures, dried in Petri dishes at ≃35°, are inoculated with two V-shaped streaks of an emulsion in sterile saline of a subculture of *Candida brumptii* (prep. described) and are incubated at 30° ± 2° for 24 hr., the growth being observed visually. A blank test is made with a sample of the same kind of meat product known to be free from preservatives, the concn. of curing salts being adjusted to about the same level as in the test sample. The level of detection is the concn. of antimicrobial agent in the meat product that gives no growth or only a few punctiform colonies, copious development being observed in the blank. The levels of detection of preservatives in ham are quoted. A. O. JONES

321. **Biphenyl and *o*-phenylphenol as preservatives for citrus fruit: a simple method for detection and determination of *o*-phenylphenol.** M. Ihloff and M. Kalitzki (Chem. u. Lebensmittel-Untersuchungs-anst., Hamburg). *Dtsch. LebensmittRdsch.*, 1960, **56** (5), 139–140.—About 4 medium-size oranges (or an equiv. amount of other citrus fruit) are disintegrated and, after addition of 500 ml of H_2O, 10 ml of H_3PO_4 and a little silicone anti-foam, the mixture is distilled from a 2-litre flask in a special return-flow apparatus for 2 hr. The ethereal oil, containing all the *o*-phenylphenol, collects in a measuring limb. A measured 1 to 2 ml of the oil is dissolved in 10 ml of light petroleum and extracted with 0·1 N NaOH (3 × 3 ml). The mixed alkaline extracts are made up to 10 ml, and aliquots (0·01 to 0·10 ml) are chromatographed (descending flow) on S. & S. 602 H/P paper, with 0·1 N NaOH as mobile phase. After 1·5 to 2 hr. the still damp paper is sprayed with a 0·5% soln. of 2,6-dibromo-*p*-benzoquinone-chlorimine in 96% ethanol. The blue spot given by *o*-phenylphenol is compared for R_F value and intensity against spots from known amounts (preferably 1 to 10 μg). Results within about 20% of theoretical values are reported. C. L. HINTON

322. **Improved method for the determination of nucleic acids in yeast.** Yoshiro Kuroiwa and Eiichi Kokubo. *Rep. Res. Lab. Kirin Brewery Co., Yokohama*, 1959, (2), 63–72 (in English).—The method previously described [Kuroiwa and Horie, *Ibid.*, 1958, (1), **33**; *Bull. Agric. Chem. Soc. Japan*, 1955, **19**, 35] has been modified by introducing an extraction with water into the procedure, to ensure complete removal of interfering substances. *Procedure*—Pressed yeast (2 g, ≃0·5 g of dry matter) is adjusted to a moisture content of 80% in a centrifuge tube, treated with 2 g of solid CO_2 and 2 ml of ethyl ether and frozen overnight at −70°. The yeast is extracted with ice-cold ethanol (4 × 20 ml), then with ethanol - ether (3 : 1) (3 × 20 ml) at 90° for 3 min., and dried in a desiccator. The residue is then extracted with 5 ml of ice-cold water for 10 min. and centrifuged for 10 min., and this process is repeated 10 times. *Determination of RNA*—The residue is suspended in 5 ml of N $HClO_4$, stored at 0° overnight and then centrifuged. Extraction for 30 min. and centrifuging are repeated 14 times; the combined extracts are diluted to 100 ml, 1 ml is diluted to 25 ml, and the extinction is read at 260 mμ in a 1-cm cell. Then RNA phosphorus (μg per g of dry yeast) = $2500x/0{\cdot}343\,w$, where x is the extinction and w is the wt. (g) of dry yeast taken. *Determination of DNA*—The residue from the RNA extraction is suspended in 5 ml of 0·5 N $HClO_4$ and extracted for 20 min. at 70°. The extraction is repeated thrice and the combined extracts are made up to 25 ml. A portion of this soln. is mixed with twice its vol. of diphenylamine reagent (Dische, *Microchem.*, 1930, **2**, 26), boiled for 5 min. and rapidly cooled, and the extinction is read after 20 min. at 600 mμ. Then DNA phosphorus (μg per g of dry yeast) = $75\,y/0{\cdot}047\,w$, where y is the extinction and w is the wt. of dry yeast taken. R. E. E.

323. **Rapid method for detailed analysis of the α-acid fraction of hops by gas chromatography.** F. L. Rigby, E. Sihto and A. Bars (Res. Div., Canadian Breweries, Ltd., Toronto, Ont.). *J. Inst. Brewing*, 1960, **66** (3), 242–249.—The dry lead salts (250 mg) of the mixed α-acids are placed in one limb of a sealed Pyrex-glass tube bent in the form of an inverted V and pyrolysed at 400° for 10 min., the fatty acids liberated from the side-chains being condensed in the other limb. The tube is then broken at the bend, and the distilled acids are esterified by adding 0·1 ml of a soln. of 2·5 ml of conc. H_2SO_4 in 100 ml of isopropyl alcohol, sealing the tube, and heating in boiling water for 1·5 hr. The tube is then cooled and opened, the contents are shaken with 0·2 ml of H_2O and 0·1 ml of light petroleum, and an aliquot of the petroleum layer is chromatographed on a column of Celite containing 10% of Ucon 50HB2000 at 50° with a helium flow-rate of 40 ml per min. Results for 13 hop samples are compared with those obtained by counter-current distribution. Some alternative procedures are discussed. R. E. E.

324. **Spectrophotometric determination of I.T.T. value of beer.** Yoshiro Kuroiwa and Jun-Ichiro Kataoka. *Rep. Res. Lab. Kirin Brewery Co., Yokohama*, 1959, (2), 43–48; *Bull. Agric. Chem. Soc. Japan*, 1960, **24** (5), 511–516 (in English).—The method described avoids the uncertainty in the visual estimation of the "80% decolorisation time" of 2,6-dichlorophenolindophenol in the usual form of the Indicator Time Test (Gray and Stone, *J. Inst. Brewing*, 1939, **45**, 253). The reagent is a 0·005 M soln. prepared from very pure 2,6-dichlorophenolindophenol (*cf.* Heyer and Paukner, *Brauwissenschaft.*, 1952, **12**, 208). The extinction of this soln. decreases slowly with time, and it should be discarded when it cannot be completely decolorised

ı ascorbic acid. *Procedure*—A "50% decolor-on standard" is prepared by mixing 0·125 ml of ent with 10 ml of acetate buffer soln. (pH 4·4), suring the extinction in a 1-cm cell at 520 mμ 25°, and calculating the extinction for 80% olorisation therefrom. The decolorisation curve eer is obtained by adding 0·25 ml of reagent to 1l of beer, measuring the extinctions at given rvals of time, and plotting the results on a h of extinction *vs.* time (in sec.). A blank curve hen obtained by taking a number of 10-ml uots of the beer, adding to each 0·25 ml of ent and maintaining them at 25°, then, at n intervals, adding to the tubes, in succession, ml of a 2% soln. of ascorbic acid in the buffer . and measuring the extinctions. The results corrected by adding to each the extinction esponding to 80% decolorisation, and the ected results are plotted on the same graph. time corresponding to the point of intersection he two curves gives the I.T.T. value. The dye vs Beer's law in beer and in water, and the nol in the beer has little influence on the results, h agree well with those obtained by the visual edure. R. E. E.

25. **Colorimetric determination of iron in wine : sulphosalicylic acid.** H. Konrad (Inst. f. ensmittelchemie beim Magistrat von Gross-in). *Nahrung*, 1960, **4** (4), 365–372.—*Pro-re*—The sample (10 to 25 ml) is evaporated in atinum dish on a water bath and then ashed 00°. The ash is dissolved in 3 ml of conc. HCl, the soln. is diluted with H_2O and transferred 50-ml flask. Sulphosalicylic acid soln. (20%) l) and 0·1 g of hydroxyammonium chloride are ed. A greenish-blue coloration after the addition xcess of aq. NH_3 indicates the presence of Cu, hich case a few drops of 10% KCN soln. must dded. The mixture is made up to volume, ed if necessary, and the extinction is measured 24 mμ; K^+, Na^+, Ca^{2+}, Cl^-, SO_3^{2-}, SO_4^{2-} and $^{5-}$ (4 g per litre), Mg^{2+} (3 g per litre) and Al^{3+} g per litre) do not interfere. Full recovery of l Fe is obtained. I. Dickinson

26. **Chromatographic analysis of glycerides. I. er chromatography of synthetic and natural erides as their mercury addition compounds.** niyuki Inoue and Manjiro Noda (Coll. of Agric., r., Kyoto). *J. Agric. Chem. Soc. Japan*, 1959, (6), 452–456.—A new method for separating erides by reversed-phase paper chromatography escribed. Twelve synthetic unsaturated mono-, and tri-glycerides containing oleic or linoleic are well separated as their mercuric acetate ucts on paper impregnated with petroleum rocarbon or tetralin. The developing solvent is nanol - acetic acid - tetralin or petroleum hydro-on, and the spots of the adducts are detected praying with a 0·2% ethanolic soln. of diphenyl-azone. The method was applied to the separa-of the triglycerides in natural fats, which give acteristic chromatograms depending upon their eride structure. The R_F value generally in-ses with increase in unsaturation of the fat.
S. Natori

27. **Method for the quantitative separation of acids from unsaponifiable matter.** H. G. mons and S. M. Wiggs (Metabolic Res. Unit, e Bromwich General Hospital, Birmingham, and). *Analyst*, 1960, **85**, 417–418.—An aliquot of the sample (*e.g.*, food, faeces or serum) is saponified with ethanolic KOH soln. and the fatty acids and unsaponified matter are extracted with light petroleum. The residue from evaporation of the extract under reduced pressure at <50° is re-extracted with light petroleum and the filtered extract is reduced to a small vol. under reduced pressure. Alumina (1 g per 25 mg of fatty acid) is added, the mixture is shaken, the supernatant liquid is filtered and the residual alumina is extracted with ethyl ether. The combined extracts are evaporated and the unsaponifiable matter is weighed. The alumina is freed from solvent and is covered with methanolic HCl and set aside overnight in a stoppered flask at 37°. The methanolic liquid is filtered, the alumina is washed with ethyl ether and the solvent and HCl are removed by evaporation under reduced pressure. The residue is extracted with light petroleum, the filtered extract is dried with Na_2SO_4, then evaporated, and the methyl esters are weighed. Recovery is 90 to 95%. The method has been applied to the preparation of the methyl esters of the fatty acids in a form suitable for analysis by vapour-phase chromatography. A. O. Jones

328. **Modified method for the paper chromatography of long-chain fatty acids.** R. Chayen and E. M. Linday (Dept. of Chem. Engng, University College, London). *J. Chromatography*, 1960, **3** (5), 503–504 (in English).—Long-chain fatty acid mixtures can be chromatographed directly by the reversed-phase ascending technique on Whatman No. 3 filter-paper impregnated with a 10% soln. of liquid paraffin in ethyl ether, with acetone - water (4:1) as solvent. The purity of the acetone is important. The time required for separation is about 3 hr. The spray reagent used is a mixture of 0·2% Nile blue (C.I. Basic Blue 12) sulphate soln. in ethanol (50 ml), triethanolamine (15 ml) and ethanol (50 ml); this gives a background colour of red with blue spots indicating the positions of the acids. A control sample must be run to compensate for the variations of R_F with temperature. S. M. Marsh

329. **Determination of chemical antioxidants in fats after separation by partition chromatography.** K. G. Berger, N. D. Sylvester and D. M. Haines (The Laboratories, J. Lyons & Co. Ltd., Kensington, London). *Analyst*, 1960, **85**, 341–345.—The chromatographic material is Silastic 181 (a synthetic rubber), with light petroleum as the stationary and aq. acetone as the mobile phase. The fat (3 to 4 g) in light petroleum soln. is applied to the column maintained at 21° by means of a water jacket. The column is washed with aq. acetone, 200 ml and then 100 ml of eluate being collected separately. In aliquots of these, butylated hydroxyanisole is determined by means of the colour produced with 2,6-dichloro-*p*-benzoquinone-4-chlorimine, the extinction being measured at 610 mμ. For determination of gallates, the eluate is treated with ferrous tartrate and sodium acetate soln. and extracted with light petroleum - isoamyl alcohol. The extinction of the extract is measured at 532 mμ for determination of the higher gallates and that of the aq. layer (diluted with acetone) at 532 mμ for propyl gallate. A test for differentiating octyl and lauryl gallates is described. To determine butylated hydroxytoluene (BHT), the soln. in 80% ethanol is treated with KOH soln., and the difference in the extinctions before and after holding the soln. at 31° for 2 hr. is a measure of the concn. of BHT.
A. O. Jones

330. **Antioxidants. The micro-determination of quinol.** M. Z. Barakat, S. K. Shehab and A. M. Abdalla (Fac. of Vet. Med., Cairo Univ., Giza, Egypt). *J. Amer. Pharm. Ass., Sci. Ed.*, 1960, **49** (6), 360–362.—To determine quinol (**I**) in oils and fats, dissolve the sample (10 g or 10 ml, containing 0·5 to 2 mg of **I**) in ethyl ether (150 ml), add a satd. aq. soln. of $NaHCO_3$ (2 ml), 4% KI soln. (5 ml) and 1% starch indicator soln. (10 drops), and titrate drop by drop with 0·1% *N*-bromosuccinimide, with constant shaking. Use the same procedure for the determination of **I** in ether anaesthetics. To determine **I** in a photographic developer which also contains interfering sulphite, add excess of $BaCl_2$ soln., shake, filter and apply the procedure to the filtrate. The experimental error is $<\pm 2\%$. The method has been successfully applied to butter, lard and cod-liver oil, for amounts of **I** as small as 0·55 mg. A. R. Rogers

331. **Colorimetric determination of thiamine by reaction with ninhydrin.** Yu. M. Ostrovskiĭ and M. A. Gvozdeva. *Byul. Eksperiment. Biol. i Med.*, 1959, **48** (11), 120–121; *Ref. Zhur., Khim., Biol. Khim.*, 1960, (9), Abstr. No. 11,776.—To 3 ml of phosphate buffer soln. add the sample soln. (first neutralised if necessary) containing 10 to 200 μg of thiamine and 0·5 ml of 0·1% ninhydrin soln. Place the mixture in a test-tube in a boiling-water bath for 20 min., cool, and dilute to 5 ml with water. The resulting yellow soln. has an absorption maximum in the short-wave region of the visible spectrum. Greatest sensitivity is achieved with a violet filter. It is shown that the formation of the coloured compound is due to the thiazole portion of the thiamine molecule. The free amino-group present in the thiamine pyrimidine ring probably does not take part in the reaction, since formaldehyde, which blocks the reaction of amino-groups with ninhydrin, does not affect the reaction with thiamine. C. D. Kopkin

332. **Separation of some vitamins of the B complex and ascorbic acid by thin-layer chromatography.** H. Gänshirt and A. Malzacher (Wissenschaftl. Abt., Fa. Dr. Karl Thomae G.m.b.H., Biberach an der Riss, Germany). *Naturwissenschaften*, 1960, **47** (12), 279–280 (in German).—The method is applicable to about 50 to 100 μg of substance containing 1 to 10 μg of the vitamin-B group and 5 to 30 μg of ascorbic acid. Plates (20 cm × 20 cm) are coated with silica gel G (Merck) to which has been added 2% of Leuchtstoff ZS-Super (Riedel de Haen) which fluoresces at 254 mμ in u.v. light. The chromatogram is developed in the dark with acetic acid - acetone - methanol - benzene (1:1:4:14). Thiamine is strongly adsorbed by the plate, the other vitamins have the following R_F values—pyridoxine 0·15, ascorbic acid 0·30, riboflavine 0·35, Ca pantothenate 0·57, nicotinamide 0·65, and biotin 0·80. Methods for the identification of the individual vitamins are given. B. Heining

333. **Determination of vitamin B_{12}. A critical review.** W. H. C. Shaw and C. J. Bessell (Glaxo Lab. Ltd., Greenford, Middx., England). *Analyst*, 1960, **85**, 389–409.—The physical and chemical methods reviewed include colorimetric methods, direct and indirect spectroscopic methods and applications of polarography and isotope dilution. It is suggested that spectrofluorimetric methods would repay further study. Methods for separation from crude materials and from analogues include adsorption chromatography, column and paper partition chromatography ion-exchange methods and paper electrophoresis. Methods for pretreatment of natural materials (*e.g.*, liver, blood and urine) in which the B_{12} may be bound are described. Microbiological assays, assays with higher animals and clinical assays are reviewed. (174 references.) A. O. Jones

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See also Abstracts—**157**, Ethanol in aq. media. **306**, Potency of isomerised vitamin-A palmitate.

Sanitation

Analysis of air, water, sewage, industrial wastes, industrial poisons.

334. **Detection and estimation of low concentrations of aldehyde in air.** E. E. Hughes and S. G. Lias (Nat. Bureau Standards, Washington, D.C., U.S.A.). *Anal. Chem.*, 1960, **32** (6), 707–708.—The aldehyde is adsorbed from the air stream on a column of silica gel, which is then treated with a mixture of *p*-phenylenediamine (**I**) and H_2O_2. The oxidation of **I** is catalysed by the aldehyde and the depth of the colour formed is a measure of the aldehyde adsorbed. Concn. as low as 10^{-8} mole % can easily be detected. Nitriles, aldehyde ammonia, oximes and aldehyde bisulphite compounds interfere, but these are not usually significant air pollutants. G. P. Cook

335. **Rapid determination of toluene and styrene vapours in the atmosphere.** A. J. Blake and B. A. Rose (D.S.I.R., Lab. of the Gov. Chemist, Clement's Inn Passage, Strand, London). *Analyst*, 1960, **85**, 442–445.—The apparatus is that previously described for the determination of benzene vapour ("Methods for the Determination of Toxic Substances in Air, No. 4: Benzene Vapour," H.M. Stationery Office, London, 1958). For determination of toluene vapour the bubbler contains 0·5 ml of formaldehyde soln. (40% w/v) diluted to 10 ml with H_2SO_4 - H_3PO_4. The number of inflations of the bulb required to produce a colour in the reagent equal to that in the standard soln. is noted and the toluene concn. (p.p.m.) is ascertained from a table. For the determination of styrene by a similar procedure, the bubbler contains 10 ml of 89·5% H_2SO_4. The standard colour soln. for toluene is a mixture of 64·3 ml of 0·1 M $CoSO_4$, 1·7 ml of M/60 $K_2Cr_2O_7$ and 34·0 ml of 0·125 M $(NH_4)_2SO_4.NiSO_4$, and for styrene 6·9 ml of M/60 $K_2Cr_2O_7$ and 4·3 ml of 0·1 M $CoSO_4$ mixed and diluted to 250 ml. The reagent for determining toluene is comparatively insensitive to benzene and 100 p.p.m. of styrene produces no colour after six inflations of the bulb. Benzene, toluene and dimethylstyrene (100 p.p.m. of each) produce no colour in the test for styrene, but vinyltoluene interferes. A. O. Jones

336. **A review of the literature of 1959 on waste water and water-pollution control. Analytical methods.** Water Pollution Control Fed. *J. Water Pollut. Control Fed.*, 1960, **32** (5), 443–452.—This review is presented of methods for determining B.O.D., C.O.D., dissolved oxygen, nitrogen compounds, sulphur compounds, halogen compounds, organic compounds and metal ions in water. (9 references.) C. A. Slater

7. Application of cellulose phosphate to the ng of high-purity water and the development of a test of high sensitivity [for copper and iron]. . Kember (National Chem. Lab., Teddington, lx., England). *Analyst*, 1960, **85**, 449–451.— the apparatus described, a disc of cellulose phate is held in a polyethylene block sur- nted by a glass funnel. For the determination u, the pH of the sample is adjusted to >4 dil. aq. NH_3 and the liquid is passed through disc. The disc is removed, placed on filter- r, exposed to NH_3 vapour and sprayed with 5% soln. of dithio-oxamide in 80% (v/v) aq. nol. The colour produced is compared with of previously prepared standards. For the rmination of Fe, the sample is acidified with (v/v) HCl and poured through the disc, which ien dipped into $K_4Fe(CN)_6$ soln., set aside for lin. on filter-paper, washed with water and pared with previously prepared standards. lower limits of detection are $0{\cdot}4 \times 10^{-9}$ part u and 2×10^{-9} part of Fe in a 50-ml sample.

A. O. Jones

8. Chromatographic separation and identifica- of organic acids. Application to detection of nic acids in river waters. H. F. Mueller, T. E. on and M. Ferretti (Illinois State Water Survey, ana, U.S.A.). *Anal. Chem.*, 1960, **32** (6), 687– —The sample is evaporated to low bulk in the ence of NaOH and after acidification the organic s are extracted into ether. The acids are ted with NaOH soln., the ether is removed by oration and the remaining aq. phase is evapor- to 1 ml, acidified, and chromatographed on a c acid column; the mobile phase consists of ous concn. of n-butanol in $CHCl_3$, and dil. D_4 is the stationary phase. The 5-ml fractions analysed by titration with NaOH soln., with nothymol blue as indicator. The recoveries >90% for all acids. The identity of some s is confirmed by comparison of the R_F values ined for pure acids by paper chromatography ne sodium salts, free acids or hydroxamates. lts for 25 samples taken from the Mississippi Ohio rivers are discussed.

G. P. Cook

9. Determination of free and total amino-acids amines in waste waters [from sugar refineries]. nkovic (CEBEDEAU, Liège, Belgium). *Bull. . Belge Étud. Docum. Eaux*, 1960, (113), 165– —Two colorimetric methods are described. *edure*—Evaporate 100 ml of sample, under ced pressure and below 40°, to a vol. of 10 ml. t (*i*), hydrolyse 5 ml of the concentrate with of conc. HCl in a sealed tube at 105° for 24 to . Dry over P_2O_5 and NaOH *in vacuo*. Dissolve residue in H_2O, centrifuge at 1200 r.p.m., and the supernatant liquid up to a vol. such that contains 10 μg of N. Determine the total e N at 485 mμ with Na 1,2-naphthaquinone- phonate (**I**) (Frame *et al.*, *Actualités Biochim.*, . **4**, 38). (*ii*) Transfer 1 ml of concentrate to ntrifuge tube, add 10 ml of 95% ethanol to pitate proteins and centrifuge at 1200 r.p.m. an aliquot containing 2·5 to 10 μg of N and rmine the free α-amino-acids at 570 mμ with ydrin (Yamagishi and Yoshida, *J. Pharm. Soc. n*, 1953, **73**, 675). Free amines and amines decarboxylated amino-acids are extracted by nuous liquid - liquid extraction for 24 to 48 ith ethyl ether and determined with **I**. Beer's s obeyed over the range 10 to 30 μg of N with od (*i*) and 2·5 to 10 μg with method (*ii*).

R. A. Hendey

340. Determination of cobalt in water. I. A. Mikhalyuk (Kiev Med. Inst.). *Lab. Delo*, 1960, **6** (3), 26–27.—For the calibration graph prepare 10-ml aliquots containing Co (as chloride or nitrate) at intervals of 0·2 μg in the region of the expected value. To each of these aliquots add H_2O (10 ml), HNO_3 (1 : 1) (0·5 ml) and HCl (1 : 1) (0·5 ml). Boil for 90 sec., add 1 ml (≡ 1 mg) of nitroso-R-salt soln. and 2 ml of fresh Na acetate soln. Boil for 45 sec., add HNO_3 (1 : 1) (2 ml) and boil for a further 45 sec. Cool and adjust the vol. to 20 ml with H_2O. Measure the extinction with a blue filter. Evaporate 1 litre of sample to dryness in a porcelain dish. Moisten the residue with H_2O, dissolve it in conc. HNO_3 (2 ml), evaporate to dryness and proceed as described above. The accuracy is within 7%.

A. Burwood-Smith

341. Possible use of ammonium 12-molybdo- phosphate for assaying certain radioactive fission products in water. R. W. C. Broadbank, S. Dhabanandana and R. D. Harding (School of Chemistry, Coll. of Technol. and Commerce, Leicester, England). *Analyst*, 1960, **85**, 365–370.—Microgram amounts of many cations, including those of the fission products ^{137}Cs - ^{137m}Ba, ^{90}Sr - ^{90}Y and ^{144}Ce - ^{144}Pr, are removed from neutral soln. by passage through a pre-formed layer of ammonium 12-molybdophosphate. Caesium is selectively retained by this compound from soln. in dil. HNO_3. To determine ^{137}Cs in rain water, the sample, acidified with HNO_3, is poured through an even layer of $(NH_4)_3PO_4.12MoO_3$ on Whatman No. 1 filter-paper in a de-mountable Buchner funnel of diameter 1 in. The filter, dried by suction, is mounted with the ppt. on an aluminium tray and counted with an end-window Geiger counter. For μg amounts of Cs no correction for self-absorption appears to be necessary. In a quoted application of the method, 1100 ml of rain water, collected in 1958, showed an activity of 12 ± 2 μμC per litre. Only relatively large amounts of Tl^+ appear to affect the removal of Cs from dil. HNO_3 soln. If present in sufficient amount Ca and Mg interfere with the assay of ^{90}Sr - ^{90}Y by this procedure, even in the presence of EDTA. Directions for the preparation of the layer of $(NH_4)_3PO_4.12MoO_3$ are given.

A. O. Jones

342. Studies on fluorescence of coumarin deriva- tives. I. Fluorimetric analysis of warfarin. Yoji Ichimura (Nat. Hygiene Lab., Tamagawayoga- machi, Setagayaku, Tokyo). *J. Pharm. Soc. Japan*, 1959, **79** (8), 1079–1082.—Warfarin was determined by means of its fluorescence in the u.v. region. The fluorescence was strongest, and fairly stable, in alkaline ethanolic soln. (pH 10) and the response was linear over the range 0·5 to 8 μg per ml.

S. Natori

See also Abstracts—**37**, **38**, Be in air. **120**, F^- in water. **382**, Detection of contaminants in air by gas chromatography.

Agricultural analysis

Soil, fertilisers, herbicides, pesticides, animal feeding-stuffs.

343. Leaf analysis as a guide to the nutrition of fruit crops. I. General principles, sampling tech- niques and analytical methods. C. Bould, E. G. Bradfield and G. M. Clarke (Dept. of Agric. and

Hortic., Long Ashton Res. Sta., Bristol, England). *J. Sci. Food Agric.*, 1960, **11** (5), 229–242.—The concept of critical nutrient levels is discussed and variations in the chemical composition of leaves due to other factors such as age and season are illustrated. In a statistical study of a particular sampling procedure, the total coeff. of variation was about 6%, of which 1·2% was due to the analytical stage. After digestion of a dried sample with H_2SO_4, Na_2SO_4 and Se, N was determined as NH_4^+, P as molybdovanadophosphate and K by flame photometry. On a further sample, digested with HNO_3 - $HClO_4$, Ca (and K) were determined by flame photometry, and Mg with Titan yellow after removal of interfering ions by ion exchange.

P. D. PARR-RICHARD

344. **Wet-combustion apparatus and procedure for organic and inorganic carbon in soil.** L. E. Allison (U.S. Salinity Lab., Riverside, Calif.). *Proc. Soil Sci. Soc. Amer.*, 1960, **24**, 36–40.—An apparatus is described in which the CO_2 evolved from the sample on acidification, oxidation and boiling under reflux is carried in a stream of air through KI soln., Ag_2SO_4 soln., conc. H_2SO_4, granulated zinc and anhydrone, and absorbed in a self-indicating absorbent and weighed. The procedure can be applied to saline soils high in Cl^- and to extracts that can be evaporated to dryness. If a blank determination yields >1 mg of CO_2 the sample should be pre-aerated. *Procedures*: (*i*) *Organic C in non-carbonate soils*—The sample is treated with conc. H_2SO_4 - 85% H_3PO_4 (3:2) and solid $K_2Cr_2O_7$, heated for 10 min. with aeration, then aerated for a further 10 min. (*ii*) *Organic C in carbonate soils*—The sample is boiled with acid $FeSO_4$ soln. (57 ml of conc. H_2SO_4 and 92 g of $FeSO_4.7H_2O$ made up to 1 litre with water), then cooled, and treated as in (*i*). (*iii*) *Organic C in soil extracts*—The extract is treated with acid $FeSO_4$ soln. and evaporated to dryness in the digestion flask, then treated as in (*i*). (*iv*) *Inorganic C in calcareous soils*—The dry sample is treated with acid $FeSO_4$ soln., boiled for 3 min. with aeration and then aerated for a further 10 min. The methods are rapid and accurate. R. E. E.

345. **Determining strontium-90 in soil by electrodialysis and ion-exchange method.** E. R. Graham (Sci., Lab., Los Alamos, N. Mex., U.S.A.). *Soil Sci.*, 1959, **88**, 11–15.—The sample (5 g or more) is placed in a membrane sac (usually filter-paper), covered with water and electrodialysed at 100 V for 12 hr. or more with a platinum-foil anode and stainless-steel cathode. The liquid, together with washings with M HCl, is treated with sufficient EDTA to make the soln. 1%, stirred and adjusted to pH 10 with NaOH. Complete chelation of Ca and Mg is checked with Eriochrome black T. The soln. is then adjusted to pH 5·5 to liberate ^{90}Sr and passed through a column of Dowex 50-X12 resin (activated by alternate washings with 5% HCl and 5% NaCl soln. followed by washing with water till free from excess of NaCl) at a rate of 8 ml per sq. cm per min. The existing ^{90}Y is then eluted with 500 ml of a soln. containing 1% of citric acid and 0·75% of EDTA, adjusted to pH 5; the completion of this elution is taken as zero time for the development of further ^{90}Y activity on the column. After a given time-interval (48 hr. or more) the ^{90}Y formed is eluted at the above rate, first with 50 ml of 5% citric acid soln. at pH 1·9 to 2·1, and then with 350 ml of 5% citric acid soln. adjusted to pH 3·8. The combined eluates are evaporated to 200 ml, treated with 1 ml of carrier soln. (30 mg of Y per ml) and sufficient solid KF to make the soln. 3 M, and the ppt. is collected on a tared filter, dried with ethanol and ethyl ether, weighed and counted. The result is referred to a graph of time *vs.* ^{90}Y activity as a percentage of the total ^{90}Sr activity. R. E. E.

346. **Radiometric determination of potassium in NPK fertilisers.** K. Martínek and J. Gottfried (Res. Inst. Inorg. Chem., Ústí Nad Labem, Czechoslovakia). *Chem. Prûmysl*, 1960, **10** (6),300–302.—The determination is based on the measurement of the natural radioactivity of ^{40}K and results were in good agreement with those obtained gravimetrically with Na tetraphenylborate, or with those by the flame-photometric method. J. ZÝKA

347. **Polarographic determination of thiourea.** V. Medonos and M. Vávrová (Inst. Chem. Technol., Prague). *Chem. Prûmysl*, 1960, **10** (5), 234–237.—Thiourea yields a polarographic wave in N H_2SO_4. The intensity of the limiting current between the dropping-mercury electrode and the S.C.E. in the range −0·2 to −0·5 V is linearly dependent on the concn. of thiourea (2×10^{-3} N to 6×10^{-3} N). Cadmium acetate (3 g per litre) is added to the supporting electrolyte to remove sulphides. Cyanamide, dicyanamide and urea do not interfere. The measured values are referred to a calibration curve. If a 0·1 N mercurous sulphate reference electrode is used, the determination can be carried out without the use of an external voltage. The average error is ±2%. The method may be used for the determination of thiourea in calcium cyanamide. J. ZÝKA

348. **Determination of nitrofurazone in feed mixtures.** A. H. J. Cross, R. A. Hendey and S. G. E. Stevens (Smith Kline and French Lab. Ltd., Welwyn Garden City, Herts., England). *Analyst*, 1960, **85**, 355–359.—The sample (≡ ≃ 1 mg of nitrofurazone) is extracted with light petroleum in a Soxhlet apparatus, the extract being rejected. The dry residue is extracted with CCl_4, the extract being again rejected. The final residue is now extracted with acetone in an apparatus protected from light. Most of the acetone in the extract is removed, 0·1 N $KMnO_4$ is added until the colour persists, the remainder of the acetone is removed, the yellowish residue is dissolved in dimethylformamide and the soln. is adjusted to a known vol. Each of two equal aliquots (≡ 0·05 to 0·35 mg of nitrofurazone) is treated with 5 ml of a 5% soln. of phenol in dimethylformamide, then one is treated with 2·5 ml of N NaOH and diluted to a known vol. with dimethylformamide, and the other (the blank) with a soln. of sodium dithionite in N NaOH and diluted with dimethylformamide. After centrifuging the soln., the extinction at 530 mμ of the sample soln. is measured against the blank and referred to a calibration graph. Recovery of known amount from complex feed mixes ranged from 99 to 101·5%. A. O. JONES

349. **Determination of captan in plant extracts.** E. Somers and D. V. Richmond (Res. Sta., Long Ashton, Bristol, England). *Analyst*, 1960, **85**, 440–442.—A suitable aliquot of a soln. of captan in benzene is evaporated almost to dryness on a water bath in a stream of dry air, the residue being allowed to dry at room temp. Pyridine - tetraethylammonium hydroxide soln. (prep. described) is added and

r 3 min. the extinction of the liquid is measured 430 mμ and referred to a calibration graph. r's law is obeyed over the range 0 to 75 μg of an. gamma-BHC, chlorobenzilate, chlorben- DDT, dieldrin and endrin do not interfere. n fruit and leaf extracts, recovery is satis- ory except in the presence of high concn. of t wax (*e.g.*, with strawberries). A. O. Jones

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e also Abstract—**351**, Soil percolator.

5.—GENERAL TECHNIQUE AND APPARATUS

General

50. **Simple and universally applicable technique handling air- and moisture-sensitive substances.** Niedermaier (Badische Anilin- & Soda-Fabrik G., Ludwigshafen/Rhein, Germany). *Z. anal. m.*, 1960, **174** (6), 407–411.—The weighing bottle ept in a stream of Ar or of specially purified N cylindrical vessel, and the sample is introduced means of a tube sealed into the stopper of the nder. If liquid, it is forced over under pressure . if solid, it is tipped in from a modified filter- e. The bulk sample is maintained in the inert osphere. The weighing bottle is then closed a ground-glass stopper on a long stem, and is ily lifted out of the cylinder by means of a form on a stem passing through a sleeve in the om of the cylinder. The apparatus is illu- ted. J. P. Stern

51. **An all-round soil percolator.** K. Gundersen anical Inst., Univ. Gothenburg, Sweden). *nce*, 1960, **132**, 224–225.—A description is n of a simple soil percolator for studies of the in a water-saturated state under aerobic condi- s. The apparatus can also be used in a closed em with a gas other than air for anaerobic eriments, and the whole percolator can be auto- ed easily for experiments with sterile soil. R. A. Hendey

52. **Fractionating column for analysis of nucleic s.** J. D. Mandell and A. D. Hershey (Carnegie . of Washington, Cold Spring Harbor, N.Y.). *l. Biochem.*, 1960, **1** (1), 66–77.—The column is ared with kieselguhr and serum albumin (*cf.* man, *Biochim. Biophys. Acta*, 1955, **18**, 132), albumin being made basic by methylation with anol. Nucleic acids are adsorbed by the mn from neutral soln. 0·4 M or less in NaCl, and eluted by higher salt concn. that are character- for the individual nucleic acids. Applications described. H. F. W. Kirkpatrick

53. **Alignment block and clamp for capillary ometers.** W. M. Robinson (BP Australia, Ltd., cenhead, S. Australia). *Bull. A.S.T.M.*, 1960,), 69–70.—Constructional details are given of an ngement which, fitted to Cannon-Fenske visco- ers, reduces the max. deviation of the calibration t. (*C*) to ±0·1% (for opaque liquids) and 0008% (for clear liquids). The holder can be oted to any other type of viscometer. W. J. Baker

354. **Pyrolysis as an analytical tool.** R. N. Rogers, S. K. Yasuda and J. Zinn (Los Alamos Sci. Lab., N. Mex., U.S.A.). *Anal. Chem.*, 1960, **32** (6), 672–678.—Pyrolysis curves of both organic and inorganic compounds are approximately the first derivative of the thermogravimetric pyrolysis curves and a new, simple type of instrument, designed to replace the thermobalance, is described. The way in which pyrolysis data may be used to obtain kinetic constants is discussed and several pyrolysis curves are presented. K. A. Proctor

355. **Thermochemical titrations in fused salts.** J. Jordan, J. Meier, E. J. Billingham, jun., and J. Pendergrast (Pennsylvania State Univ., University Park, U.S.A.). *Anal. Chem.*, 1960, **32** (6), 651–655.—Chloride has been determined at concn. between 8×10^{-4} and 2×10^{-2} M in a molten $LiNO_3$ - KNO_3 eutectic at 158° by a thermometric precipitation method, with a standard soln. of $AgNO_3$ in the eutectic as titrant. The titrations were performed automatically by remote control in an adiabatic cell operated at 150° to 200°, in which random temp. fluctuations were reduced to ±0·0005°. Temperature changes during the titration were monitored by means of a thermistor bridge and automatically recorded. The method appears to be generally applicable to the rapid and convenient determination of chloride in fused nitrate melts. K. A. Proctor

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See also Abstracts—**14**, High-temp. vacuum furnace for thermal analysis. **151**, Apparatus for H in hydrocarbons.

Chromatography, ion exchange, electrophoresis

356. **An extension of the gradient-elution principle.** D. G. O'Sullivan (Courtauld Inst. Biochem., Middlesex Hosp. Med. School, London). *Analyst*, 1960, **85**, 434–440.—Methods of eluting chromatographic columns with more than one solvent are discussed mathematically. In the "constant volume" method, as each drop of the first solvent falls on the column from the reservoir an equal drop of the second falls into the reservoir. For greater flexibility in this type of concn. gradient, the second solvent is added to the first at a rate different from that at which the first solvent feeds the column. This can be effected by connecting two cylindrical reservoirs of different cross-sectional area by a siphon, and arranging the flow to the column so that the levels of liquid in the two reservoirs fall at equal rates. Both the constant-volume method and the siphon method are extended to three eluting solvents and, in addition to the mathematical treatment, graphs of the concn. of the second and third solvents in the mixed eluting liquid plotted against the volume of eluting liquid leaving the mixing apparatus are shown. A. O. Jones

357. **Technique for simultaneous multiple-column gradient-elution chromatography.** P. Vestergaard (Rockland State Hospital, Orangeburg, N.Y., U.S.A.). *J. Chromatography*, 1960, **3** (6), 560–569.—A variable-speed drive operates a syringe which delivers one liquid to a mixing chamber. This feeds eluent to a number of columns in parallel so that the same gradient is used for a set of standards

and unknowns. The gradient can be changed during a run by altering the speed of the syringe drive. A. R. ROGERS

358. **Optimisation of resolution - time ratio with packed chromatographic columns.** R. J. Loyd, B. O. Ayers and F. W. Karasek (Res. and Devel. Dept., Phillips Petroleum Co., Bartlesville, Okla., U.S.A.). *Anal. Chem.*, 1960, **32** (6), 698–701.—The effects of the quantity of stationary phase and the choice of carrier gas on the resolution - time ratio obtained with packed gas-chromatographic columns have been investigated. Considerable improvement in the time required for a separation can be achieved by using a small proportion of stationary phase and a carrier gas of low viscosity and high diffusivity. K. A. PROCTOR

359. **A multicollector: a fraction collector for the simultaneous collection of fractions from a number of chromatographic columns.** P. Vestergaard (Rockland State Hospital, Orangeburg, N.Y., U.S.A.). *J. Chromatography*, 1960, **3** (6), 554–559.—The collector (described and illustrated) is inexpensive and easy to build. A. R. ROGERS

360. **Automatic recording elution analyser for organic acids.** Chie Yamazaki, Nobuya Nagashima and Tadao Takenishi (Central Res. Lab., Ajinomoto Co., Inc., Kawasaki, Kanagawa, Japan). *Anal. Chem.*, 1960, **32** (6), 733–734.—In the method described, the effluent from an elution-chromatographic column flows continuously through a 1-mm cell in an i.r. spectrograph and the extinction at 1715 cm^{-1} is recorded. Modifications to a Perkin - Elmer spectrophotometer Model 21 to enable it to be used for this purpose are described. K. A. PROCTOR

361. **Apparatus for collecting and drying organic acid fractions.** J. H. Schwartz (Eastern Reg. Lab., Agric. Res. Service, U.S. Dept. of Agric., Philadelphia, Pa.). *J. Chromatography*, 1960, **3** (5), 491–493 (in English).—The attachment described can be used on a Technicon fraction collector for collecting organic acid fractions that have to be subsequently evaporated and titrated. The rack holds 25 mm × 50 mm shell vials, which are more suited to the purpose than test-tubes, and the method of construction is detailed. Also described are special racks holding 10 vials which can be fastened together to form larger units and then placed in a special drying chamber for evaporation. S. M. MARSH

362. **PTFE and glass pump: chromatographic applications.** A. C. Arcus (Nutrition Res. Dept., Med. Sch., Dunedin, New Zealand). *J. Chromatography*, 1960, **3** (5), 411–414 (in English).—A detailed description is given of a pump with a glass cylinder and PTFE (polytetrafluoroethylene) piston. The piston has a sealing ring which ensures that no solvent leaks back. Chromatographic applications include the supplying of solvent under pressure or at constant rate, the de-gassing of solvents and gradient elution. S. M. MARSH

363. **A new apparatus for paper-chromatographic analyses in an inert atmosphere.** P. Szarvas, T. Balogh and L. Máczay. *Magyar Kém. Foly.*, 1960, **66** (4), 137–139.—The separation of Ti^{III} from Ti^{IV} and of V^{II} from V^{III}, V^{IV} and V^{V} has been studied. As Ti^{III} and V^{II} are readily oxidised in air, the procedure is carried out in an apparatus (described and illustrated) in which the preparation of th lower-valency ion, its application to the paper, an the drying and development of the chromatogra are carried out in an inert atmosphere. The redu tion is performed by means of nascent hydrogen f a Jones-type reductor that forms part of the dro ping device for loading the chromatogram. R. E. E.

364. **Radial-development chromatography. I** L. Peyron (Lab. de Recherches, Éts. A. Chiri Grasse, France). *Bull. Soc. Chim. France*, 196 (6), 1243–1247.—This bibliography of publication mainly from 1957 onwards, is subdivided accordin to (*i*) elution procedures, and (*ii*) separation organic and inorganic compounds. A few of th references are annotated briefly. The usefulnes of radial chromatography in the analysis of essentia oils and their constituents is stressed. (*Cf. Anal Abstr.*, 1959, **6**, 1578.) (222 references.) W. J. BAKER

365. **Improved Zaffaroni-type solvent system fo paper chromatography.** J. C. Touchstone and M Kasparow (Dept. of Obstetrics and Gynecology Sch. Med., Univ. Pennsylvania, Philadelphia) *Anal. Biochem.*, 1960, **1** (1), 91–93.—A solven system consisting of methylcyclohexane and di methylformamide, suitable for the separation o steroids not resolved by other systems, is described Details are given of the separation of 17-hydroxy deoxycorticosterone from corticosterone and fro 11-deoxycorticosterone. R. A. BRENAN

366. **An apparatus for elution from paper chrom atograms.** M. J. Canny (Botany School, Cam bridge, England). *J. Chromatography*, 1960, (5), 496–497 (in English).—The apparatus can b used for eluting large pieces of paper with smal volumes of volatile solvents, and consists essentiall of a reservoir with a paper wick, inside a glass tub which holds the paper to be eluted. The assembl is cheap and quickly constructed, only the minimu of supervision is necessary and large numbers o papers can be eluted simultaneously. S. M. MARSH

367. **Automatic solvent-addition funnel for pape chromatography.** B. L. Van Duuren (Dept. o Ind. Med., New York Univ., U.S.A.). *Anal. Chem.* 1960, **32** (6), 732–733.—The funnel described make it possible to start the running of a chromatogra automatically after a pre-determined equilibratio period, and can be used for both ascending an descending chromatography. K. A. PROCTOR

368. **Paper chromatography: multiple-spot forma tion by pure substances.** A. H. Beckett, M. A Beaven and A. E. Robinson (Chelsea Coll. Sci. an Technol., London). *Nature*, 1960, **186**, 775–776.— A study of amines and amine salts, with acid an neutral solvent systems, has shown that double spot formation is dependent on the relative R_F values of individual acids and bases of the salt, th relative concn. of the acid and base, and the relativ dissociation constants of the acids present in th salts and in the solvent system. The conditions fo multiple-spot formation are likely to occur wit amines in biological extracts that contain organi acids, or to which trichloroacetic acid has bee added, and in the investigation of the metabolis of basic drugs if strongly acid compounds are als products of metabolism. A. C. R. HARTLEY

9. **Rapid estimations of zone content in paper natographs. VI. R.LF. method.** G. G. Blake t. of Chem., Sydney Univ., Australia). *Anal. . Acta*, 1960, **22** (6), 546–548 (in English).—zone is cut out and extracted with a suitable nt (*e.g.*, water) and the conductance is ured by a rectified low-frequency (R.LF.) od (*Ibid.*, 1955, **13**, 394). The amount of solute nt is read from a calibration curve prepared standard soln. The suitable range of solute n. is 0·002 N to 0·00016 N, but for solute concn. part in 10^4 the electrostatic discharge method ., 1959, **21**, 545) is preferred. R. M. S. Hall

0. **Certain aspects of bromophenol blue staining ced from spot tests on filter-paper.** G. G. Sel- (Inst. of Animal Genetics, Edinburgh, Scot- . *J. Chromatography*, 1960, **3** (6), 531–535.—rent ways of using bromophenol blue for stain- proteins on filter-paper have been compared. sitive reaction may be given by thiols, thiamine some imidazole derivatives. A. R. Rogers

1. **Spotting apparatus for the application of e quantities of fluids on paper chromatograms.** Logothetis (Univ. of Minnesota Med. Sch., neapolis, U.S.A.). *J. Chromatography*, 1960, **3** 488–491 (in English).—The apparatus transfers specimens from eight syringes by continuous ejection on to chromatographic paper while the ressure is reduced under the spotting area by an stable vacuum-pump device; this ensures l evaporation of the drops to give deposits of material covering a small uniform area. Full tructional details are given, with diagrams. S. M. Marsh

2. **Quantitative radioassay of paper chromato- as by liquid scintillation counting. Application rbon-14-labelled salicylic acid.** W. F. Bousquet]J. E. Christian (Bionucleonics Dept., Purdue ., Lafayette, Ind., U.S.A.). *Anal. Chem.*, , **32** (6), 722–723.—The procedure described is for the quant. assay of ^{14}C-labelled salicylic on paper chromatograms. An acid scintillator m is used and eliminates the spurious count observed in non-acid scintillation media. K. A. Proctor

3. **Isolation and determination of volatile com- ds by adsorption on charcoal.** J. H. Dhont and eurman (Central Inst. for Nutrition and Food T.N.O., Utrecht, The Netherlands). *Analyst*, **85**, 419–422.—The apparatus used is described. vapour of the sample is conveyed by a current into a weighed amount of charcoal, which is re-weighed and placed on the top of a char- column. The steam-jacketed column is heated 00° and dried by passage of a stream of N. stream of N is then saturated with dioxan ur and directed through the column into catharometer. Desorption is followed by the nometer, the first deflection indicating the nce of the displaced compound and the second reak-through of the displacing dioxan. The le vapour is condensed in a U-tube cooled with ce and acetone, its vol. is measured at room ., and an aliquot is analysed by vapour-phase natography, a 3-metre column of Carbowax at 70° being used and the results plotted by an natic recorder. The ratio of the peak height ioxan to the peak height of the desorbed ound is calculated and compared with those romatograms of mixtures of known amounts of n and the compound being investigated. A. O. Jones

374. **Portable carbon dioxide-conversion apparatus for gas - liquid chromatography.** M. C. Simmons, L. M. Taylor and M. Nager (Houston Res. Lab., Shell Oil Co., Houston, Tex., U.S.A.). *Anal. Chem.*, 1960, **32** (6), 731–732.—A compact, portable CO_2-conversion unit for attachment to gas chromatographs is described. It allows the separated components to be detected as hydrocarbon bands which can be trapped for positive identification or more detailed analysis by other methods. Alternatively, hydrocarbons emerging from the chromatograph may be directed into the conversion unit, converted into CO_2, and detected with a separate thermal-conductivity detector. K. A. Proctor

375. **Coiled columns and resolution in gas chromatography.** J. C. Giddings (Dept. of Chem., Univ. of Utah, Salt Lake City, U.S.A.). *J. Chromatography*, 1960, **3** (6), 520–523.—With certain assumptions it is shown that the increase in plate height due to bending is equal to $7vr^4/48R^2D$, where v is the linear gas velocity, r is the tube radius, R is the radius of curvature, and D is the coeff. of lateral diffusion. The effect is marked with preparative-scale columns. A. R. Rogers

376. **Simple accessory to a commercial gas chromatograph for micro-catalytic studies.** L. S. Ettre and N. Brenner (The Perkin-Elmer Corp., Norwalk, Conn., U.S.A.). *J. Chromatography*, 1960, **3** (6), 524–530.—The gas-sampling valve of a commercial gas chromatograph is replaced by a combined sampler and micro-reactor. This makes possible the analysis of the products of either periodic or flow-type reactions. A. R. Rogers

377. **Relative detector response in gas chromatography. [I.]** G. R. Jamieson (Chem. Dept., Paisley Tech. Coll., Scotland). *J. Chromatography*, 1960, **3** (5), 464–470 (in English).—The relative response of thermal-conductivity detectors to a wide range of organic compounds has been investigated, with N as the carrier gas. Three comparison compounds were chosen, *viz*, benzene up to 95°, *p*-cymene at 160° and methyl palmitate at 210°. Tabulated results show that there is an increase in relative response with increasing molecular weight through a homologous series, and that for isomeric compounds there is a decrease in relative response with an increase in chain-branching. Relative response does not vary if the flow rate of N is varied within narrow limits, but is temperature-dependent. As more data become available, relative detector response might be used in conjunction with retention-volume data for identification purposes. S. M. Marsh

378. **Effect of an argon - nitrogen carrier-gas mixture on the sensitivity of a gas-chromatographic ionisation detector.** D. Welti and T. Wilkins (Unilever Ltd., Food Res. Dept., Colworth House, Sharnbrook, Bedford, England). *J. Chromatography*, 1960, **3** (6), 589–591.—The sensitivity of the "argon detector" can be varied continuously over a wide range by use of a mixture of Ar and N as carrier gas, and a conventional detector can be used with preparative-scale columns. The greatest decrease in sensitivity occurs as the proportion of N is increased from 0 to 10%. Flow-rate settings are critical for good reproducibility. A. R. Rogers

379. **Thermal conductivity cell response and its relationship to quantitative gas chromatography.** R. L. Grob, D. Mercer, T. Gribben and J. Wells (Chem. Dept., Wheeling Coll., W. Va., U.S.A.).

J. Chromatography, 1960, **3** (6), 545–553.—Analysis of blends of alcohols has shown that experimentally measured response values should be used to relate peak areas with wt. of components; the errors are <1% but, if the relative areas are used as a measure of wt. (%) on the assumption of direct proportionality, the errors are as high as 10%.

A. R. Rogers

380. **Compensated Wheatstone's bridge circuit for gas-chromatographic katharometry.** A. B. Littlewood (Dept. Chem., Kings Coll., Univ. of Durham, Newcastle upon Tyne, England). *J. Sci. Instrum.*, 1960, **37** (6), 185–188.—Suitable shunt and series resistances included in the katharometer arms of the Wheatstone bridge circuit considerably reduce base-line drift caused by imperfect temp. or voltage control. Equations are given from which the values of these resistances can be calculated from the observed drift in their absence.

G. Skirrow

381. **Response of the β-ray ionisation detector to unesterified lower fatty acids in gas - liquid chromatography.** C. J. F. Böttcher, G. F. G. Clemens and C. M. van Gent (Dept. of Phys. Chem., Univ., Leiden, Netherlands). *J. Chromatography*, 1960, **3** (6), 582–584.—The relative peak areas per unit mass of the straight-chain fatty acids (from C_4 to C_{10}) increase with molecular weight up to about 150. The relative peak area per mole is a linear function of the log of the number of carbon atoms.

A. R. Rogers

382. **A Tesla discharge detector for gas chromatography.** J. C. Sternberg and R. E. Poulson (Kedzie Chem. Lab., Michigan State Univ., East Lansing, U.S.A.). *J. Chromatography*, 1960, **3** (5), 406–410 (in English).—A laboratory Tesla coil leak tester has been used in the construction of two simple cheap high-sensitivity detectors for gas chromatography. Variations in gas composition may be detected by measuring either the emitted light intensity, or a direct-current signal produced by inserting a pair of probe-electrodes asymmetrically into the discharge. Both means of detection can be incorporated into a single detector. Air may be used as the carrier gas, and the detector could be employed for the measurement of humidity and vapour pressures above solvents or solutions, mol. wt. determinations, and the detection of atmospheric contaminants.

S. M. Marsh

383. **An apparatus for paper ionophoresis.** S. Lovett (Microbiol. Res. Estab., Porton, Wilts., England). *Chem. & Ind.*, 1960, (25), 709–710.—The apparatus described has been designed to overcome the staining obtained when copper-wire electrodes are used in electrophoresis. Provision has also been made for the simultaneous development of several electropherograms.

K. A. Proctor

384. **Simple apparatus for the application of specimens in continuous paper electrophoresis.** W. Matthias (Inst. f. Pflanzenzüchtung, Dtsch. Akad. der Landwirtschaftswissenschaften, Quedlinburg, Germany). *J. Chromatography*, 1960, **3** (5), 501–502 (in German).—A detailed description, with circuit diagram, is given of the apparatus, which is based on the principle of the oxy-hydrogen gas voltammeter.

S. M. Marsh

385. **New type of tank for paper micro-electrophoresis of proteins from biological fluids.** A. Seniów (Dept. of Parasitol. and Parasitic Diseases, High School of Agric., Wroclaw, Poland). *Chromatography*, 1960, **3** (6), 586–588.—The electrode vessels are slotted cylinders which can rotated horizontally about their axes. The wett paper strips can easily be stretched between t vessels before the samples are applied.

A. R. Rogers

386. **Apparatus for the direct photometry of lip electropherograms.** R. Ya. Vÿsotskiĭ and V. Baumgart. *Voprosÿ Med. Khim.*, 1959, **5** (5), 37 380; *Ref. Zhur., Khim., Biol. Khim.*, 1960, (Abstr. No. 8867.—The apparatus is based on t use of reflected light for estimating the colo intensity of the electropherograms. The lig absorption is measured with a photo-conducti element, which has a number of advantages over conventional photo-cell. A check of the readin from the apparatus against the amount of lipi has shown that direct photometry of the electr pherograms with a small range of colour intensiti is possible. The results of determinations sho good agreement with those of the elution method

C. D. Kopkin

387. **Starch electrophoresis. III. Starch-g electrophoresis.** H. Bloemendal (Dept. of Biochem Netherlands Cancer Inst., Amsterdam). *J. Chrom atography*, 1960, **3** (6), 509–519.—A review is pr sented of methods and applications, with references (*cf. Anal. Abstr.*, 1959, **6**, 5035; 1960, 4070).

A. R. Rogers

388. **Effects of field strength and ionic strength o velocity and spread of zones in starch-block electr phoresis.** G. L. Miller (Quartermaster Research an Engng Center, Natick, Mass., U.S.A.). *J. Chrom atography*, 1960, **3** (6), 570–575.—Fields that a too high and ionic strengths that are too low ma cause disturbances in starch-block zone electr phoresis. With the apparatus used, best result were obtained with fields of <7·5 V per cm and a ionic strengths of >0·1. There is no significan direct relation between spread of zones and diffusio or distance of migration.

A. R. Rogers

See also Abstracts—**123**, Un-impregnated cellulo paper for anions. **177**, Relative detector respon in gas chromatography. **397**, Application of i analysis to gas-chromatographic fractions.

Optical

389. **The Ebert spectrograph.** Anon. *Hilger J* 1960, **6** (2), 29–32.—Comparison of the Ebert a the Wadsworth spectrographs, in the light of rece modifications to the Ebert mounting, show t Ebert to be almost equivalent to the Wadsworth performance, and superior in that it is mechanical simpler, has a greater wavelength range and c be adapted for direct reading. The instrume illustrated is a grating spectrograph of high defi tion and resolution, capable of analysing su materials as high-alloy steels, uranium and tra uranic elements. The overlapping high-or spectra are separated by the "order sorter" d cribed.

E. G. Cummins

390. **Spectrographic electrodes for refract samples.** M. Slavin (Chem. Dept., Brookhaven N Lab., Upton, Long Island, N.Y., U.S.A.). *Ap Spectroscopy*, 1960, **14** (3), 82.—The refract components, particularly CaO and Al_2O_3, of sample tend to be lost by mechanical ejection of

ıal bead by the arc stream. A standard drill rms a cup that is too shallow and it is claimed a deep 30° conical crater, cut with a flat er, will hold the bead until it is completely ilised into the arc discharge, thereby giving improved reproducibility for these elements. P. T. Beale

. **Controlled-atmosphere excitation chamber for ırc analysis.** M. P. Brash and J. P. Phaneuf and Advanced Dev. Div., Avco Corp., Wilton, Mass., U.S.A.). *Appl. Spectroscopy*, **14** (3), 80–81.—Cyanogen bands and heavy ground are eliminated by arcing in an argon - en atmosphere. The construction of a suitable cz vessel for arcing in a nitrogen-free atmosphere scribed in detail. P. T. Beale

2. **The use of scatter diagrams in emission roscopy.** G. Holdt and A. Strasheim (Max-tk-Inst. für Metallforschung, Stuttgart, Ger-r). *Appl. Spectroscopy*, 1960, **14** (3), 64–72.—correlation and regression of two variables, as the internal standard and analysis lines for alculation of an intensity ratio, may be best n by plotting one against the other. The tical information is derived from the resulting er diagram and from a contour ellipse, con-ted so that 10% of the points fall outside or on llipse perimeter. Many worked examples of pplication of the method to different spectro-ic problems are given. P. T. Beale

3. **The additive standard method in flame rophotometry, with special reference to the ne earths.** G. E. Harrison (Radiobiological Unit, Harwell, Berks., England). *Photoelect. r. Gr. Bull.*, 1959, (12), 327–328.—A Unicam spectrophotometer is used to determine Ca, a and Mg in biological material. The inter-ce effects of the large amounts of Na, K and P nt are overcome by making standard additions e elements being determined to the sample An example is given of the determination of n a bovine serum. P. T. Beale

. **Photo-electric method for the measurement of ra of light sources of rapidly varying intensities.** . Seliger (McCollum-Pratt Inst., Johns Hopkins ., Baltimore, Md.). *Anal. Biochem.*, 1960, **1** 50–65.—The method depends on the use of multiplier tubes. Application is made to urement of the luminescence of luminol (2-phthalhydrazide), and of H_2O_2 in the presence H. F. W. Kirkpatrick

5. **Improved adaptation of Beckman model DU rophotometer for use with micro-litre absorption** D. Glick and L. J. Greenberg (Dept. of iol. Chem., Univ. of Minnesota, Minneapolis, A.). *Anal. Chem.*, 1960, **32** (6), 736.—Improve-s in certain details of an adaptation of the man DU spectrophotometer for use with o-litre absorption cells, reported previously c and Grunbaum, *Ibid.*, 1957, **29**, 1243), are ibed. K. A. Proctor

3. **Ultra-violet spectroscopy with polarised** F. Dörr and M. Held (Phys.-chem. Inst. der . Hochsch., München, W. Germany). *Angew.* ., 1960, **72** (9), 287–294.—This review of the ods of steric orientation of particular electron ators in organic molecules stresses the addi-l structural information that is made available. methods described are not confined to crystal studies. Work with otherwise orientated molecules, either confined in thin films, as with polymethine-cyanine on a poly(vinyl alcohol) foil, or as a streaming layer between glass plates (*e.g.*, pseudo-isocyanine and tobacco mosaic virus), or aligned by molecular attraction in a monomolecular layer, as with hydrophobic and hydrophilic groups, is included. External electric and magnetic fields have also been used to produce orientation and thus facilitate the measurement of the alignment of electron oscillators with the aid of polarised light. The application of the method to smaller molecules is made simpler by statistical consideration of the number of similarly orientated molecules in a solid solution (*e.g.*, ice), a state in which they are orientated at random. Both photochemical and fluorescence studies have produced results with this approach. (88 references.) E. G. Cummins

397. **Micro-quantitative infra-red analysis: application to gas-chromatographic fractions.** J. E. Stewart, R. O. Brace, T. Johns and W. F. Ulrich (Beckman Instruments Inc., Fullerton, Calif., U.S.A.). *Nature*, 1960, **186**, 628–629.—Two i.r. measuring techniques for 20 to 100-μg amounts of material are described. (*i*) In the transmittance-ratio method for two-component mixtures, the transmittance is measured at two appropriate wavelengths and the log of the ratio is plotted against concn. of one component. (*ii*) In the extinction-ratio method, the ratio of the extinction at two wavelengths is plotted against the ratio of concn.; provided that there is no band overlap, the plot is rectilinear. Close control of cell thickness, solvent concn. or v.p. is unnecessary. The application of the methods to mixtures of *m*- and *p*-xylene, *m*- and *p*-cresol, 2,6-xylenol and *o*-cresol, and anthracene and phenanthrene are described in detail. G. S. Roberts

398. **Potassium bromide capillary cell for infra-red micro-spectroscopy.** E. D. Black (Pioneering Res. Div., Quartermaster Res. and Engng Center, Natick, Mass., U.S.A.). *Anal. Chem.*, 1960, **32** (6), 735.—A method of preparing capillary cells by the potassium bromide pelleting technique, by embedding a wire in the pellet and then withdrawing it, is described. Such cells have good transmission from 2 to 15 μ. If necessary the sample may be recovered by dissolution of the pellet in water and solvent extraction. K. A. Proctor

399. **Effects due to chemical state of the samples in X-ray emission and absorption.** P. D. Zemany (Res. Lab., General Electric Co., Schenectady, N.Y., U.S.A.). *Anal. Chem.*, 1960, **32** (6), 595–597.—Measurements made with a conventional X-ray spectrograph show that the chemical state of an element can affect the structure and position of an absorption edge and both the wavelength and the intensity of an emission line. These effects may be useful in determining the valency state of an element. K. A. Proctor

400. **Electron-probe methods of X-ray micro-analysis.** P. Duncumb (Cavendish Lab., Cambridge, England). *Brit. J. Appl. Phys.*, 1960, **11** (5), 169–176.—Applications are described of the electron probe as a point source of X-rays for analysis by absorption, fluorescence and emission spectroscopy. For the examination of surfaces for which emission spectroscopy is the most suitable method, the probe excites the specimen itself. With this method a resolving power of 1 μ and a detection sensitivity

of 10^{-14} g are attainable, and a scanning technique based on a selected emission line enables the distribution of a particular element to be presented visually. G. SKIRROW

401. **New automatic X-ray fluorescence spectrometer.** J. R. Stansfield (Hilger & Watts, 98 St. Pancras Way, London, England). *Hilger J.*, 1960, **6** (2), 23–28.—Planned programming by patchboard, for the determination of up to 20 elements, is possible with the instrument described. The choice of a sequential system has made pulse-height analysis economical and therefore digital to analogue conversion is unnecessary. The choice of the many independently adjustable parameters provided, together with the selection of a patchboard to programme the angle of analysis to the nearest fiftieth of a degree, and other factors, permits automatically typed-out intensities to be presented in tabular form as they are measured. It is emphasised that the method is most suitable for the determination of major constituents and that, for example, different types of steel will need different calibration curves. Comparison with the direct-reading u.v. spectrograph is discussed. The X-ray calibration curve for nickel is a straight line through the origin with a standard deviation of 0·045% over the 8 to 16% range. E. G. CUMMINS

402. **Photo-electric automatic polarimeters.** A. Emmerich. *Zucker*, 1960, **13**, 148–153.—The determination of sucrose with the photo-electric polarimeter and the standardisation of the polarimeter are described. Owing to rotation dispersion, the reading by yellow - green mercury light (5461 A) with the normal sugar soln. is 0·18% higher than with the normal control quartz plate. Preliminary results for the ratios between readings with yellow sodium light and with yellow - green mercury light for soln. of several raw sugars and molasses indicate an increase in the rotation dispersion of the molasses. Three automatic polarimeters are described. SUGAR IND. ABSTR.

403. **The ETL-NPL automatic polarimeter, type 143A.** E. F. Dawson (Ericsson Telephones Ltd., Basford, Notts., England). *Photoelect. Spectr. Gr. Bull.*, 1959, (12), 315–320.—The split-field of the conventional instrument is replaced by a single field, but the half-shade principle is virtually retained by time-sharing by use of a Faraday cell, in which the twist in the plane of polarisation of light passing through the dense glass core is directly proportional to the current produced in the surrounding solenoid. A photomultiplier and feed-back principles in the electronic circuits have been used to produce an automatic instrument with a range of $\pm 0{\cdot}5°$ of arc and a minimum detectable rotation of one ten-thousandth of a degree. P. T. BEALE

Electrical

404. **A semi-automatic polarograph.** G. W. Drake and C. B. Johnston (Univ. of Houston, Texas, U.S.A.). *J. Chem. Educ.*, 1960, **37** (5), 240–242.—With the instrument described, the effect of voltage change is plotted manually point by point on a chart from readings on a micro-ammeter. As the chart and voltage changer are coupled, voltage calibration is straightforward and operation simple. All the parts are readily available. E. G. CUMMINS

405. **Polarographic determination of high conc trations of elements.** L. N. Lyubimova and V. Sochevanov. *Zavod. Lab.*, 1960, **26** (6), 703–707 Polarography of high concn. (0·005 to 0·25 M) c be carried out without loss of accuracy if the elec cal resistance of the mercury in the capillary reduced sufficiently. The capillary electro described consists of a short length of capilla tubing (10 to 12 mm) fused to a wider tube carryi a platinum contact. Above the contact is fuse longer capillary tube to give the necessary me anical resistance to the flow of mercury. Appli tions to the determination of Cu, Pb and Zn in o are described. G. S. SMITH

406. **pH measurement at low temperatures usi modified calomel and glass electrodes.** L. van d Berg (Div. of Appl. Biol., Nat. Res. Coun Ottawa, Canada). *Anal. Chem.*, 1960, **32** (6), 62 631.—Modified reference and glass electrodes a described, suitable for pH measurements at tem down to −30°. Polyhydric alcohols (or some their derivatives) are added to the satd. KCl so of the calomel electrodes; the glass membranes the glass electrodes are enlarged, and the inn buffer is replaced by mercury. Some prelimina results of measurements on frozen salt soln., mi and meat are given. K. A. PROCTOR

407. **Differential electrolytic potentiometry. I An examination of the variables of the meth applied to inactive reductants.** E. Bishop (Washin ton Singer Lab., The University, Exeter, Englan *Analyst*, 1960, **85**, 422–431.—A study is made of t electrical variables in the differential electroly potentiometry of irreversible reductants, *i.* applications in which the electrolytic anode inactive. For stated reasons the determination hydrazine with $KBrO_3$ was selected as a test rea tion. The effects of isolating individual electroly electrodes, varying the current with fixed electro area, reducing the size of the electrodes to the mic and ultra-micro scale, varying the electrode ar with fixed current and varying the source potenti and stabilising series resistance of the current a examined. Experimental design criteria are form lated for the application of differential potenti metry to reactions of the type studied. A. O. JONES

408. **New approach to radioassay of aqueous sol tions in the liquid scintillation spectrometer.** Steinberg (Nat. Heart Inst., Nat. Inst. of Healt Bethesda, Md.). *Anal. Biochem.*, 1960, **1** (1), 23–3 —The method described is based on a two-pha system, the solid phase consisting of finely divid phosphor and the liquid phase containing the rad active material to be assayed. A number of so phosphors, including a plastic scintillator, have be investigated and highly purified anthracene appe to be the most effective. The sample soln. pipetted on to crystals of anthracene in a standa counting bottle and the fluorescent emission counted in a liquid scintillation spectrometer. T method is applicable to virtually any compou soluble in water, salt soln., dil. acid or dil. alk and as no chemical conversion of the sample required it can be recovered unaltered for otl investigations. R. A. BRENAN

See also Abstract—**263**, Carboxymethylcellul gel for electrophoresis.

ABBREVIATIONS

Certain abbreviations in everyday use are not included in the following list. When any doubt might arise from the use of an abbreviation or symbol the word is printed in full.

alternating current	a.c.
ampere	amp.
Ångström unit	A
anhydrous	anhyd.
approximate, -ly	approx.
aqueous	aq.
atmospher-e, -ic	atm.
boiling-point	b.p.
British thermal unit	B.Th.U.
calorie (large)	kg-cal.
calorie (small)	g-cal.
centimetre	cm
coefficient	coeff.
Colour Index	C.I.
concentrated	conc.
concentration	concn.
constant	const.
crystalline	cryst.
crystallised	cryst.
cubic	cu.
current density	c.d.
cycles per second	c/s
density	ρ
density, relative	d or wt. per ml
dilute	dil.
direct current	d.c.
distilled	dist.
ethylenediaminetetra-acetic acid	EDTA
electromotive force	e.m.f.
equivalent	equiv.
gram	g
gram-molecule	mole
half-wave potential	$E_{\frac{1}{2}}$
hour	hr.
hydrogen ion exponent	pH
infra-red	i.r.
insoluble	insol.
international unit	i.u.
kilogram	kg
kilovolt	kV
kilowatt	kW
liquid	liq.
maxim-um, -a	max.
melting-point	m.p.
microgram	μg (not γ)
microlitre	μl
micromole	μmole
micron	μ
milliampere	mA
milli-equivalent	milli-equiv.
milligram	mg
millilitre	ml
millimetre	mm
millimicrogram	mμg
millimolar	mM
millivolt	mV
minute (time)	min.
molar (concentration)	M
molecul-e, -ar	mol.
normal (concentration)	N
optical rotation	α_λ^t
ounce	oz
parts per million	p.p.m.
per cent.	%
per cent. (vol. in vol.)	% (v/v)
per cent. (wt. in vol.)	% (w/v)
per cent. (wt. in wt.)	% (w/w)
potential difference	p.d.
precipitate (as a noun)	ppt.
precipitated	pptd.
precipitating	pptg.
precipitation	pptn.
preparation	prep.
qualitative, -ly	qual.
quantitative, -ly	quant.
recrystallised	recryst.
refractive index	n_λ^t
relative band speed	R_F
relative humidity	r.h.
revolutions per minute	r.p.m.
saponification value	sap. val.
saturated calomel electrode	S.C.E.
second (time)	sec.
soluble	sol.
solution	soln.
specific gravity	sp. gr.
specific rotation	$[\alpha]_\lambda^t$
square centimetre	sq. cm
standard temp. and pressure	s.t.p.
temperature	temp.
ultra-violet	u.v.
vapour density	v.d.
vapour pressure	v.p.
volt	V
volume	vol.
watt	W
wavelength	λ
weight	wt.

In addition, the following symbols may be used in conjunction with numerical values or in mathematical expressions—

greater than	$>$	less than	$<$
not greater than	$\ngtr$	not less than	$\nless$
is proportional to	$\propto$	of the order of, approximately	$\simeq$

The principal Pharmacopoeias are denoted by B.P., U.S.P. or D.A.B., together with the identifying roman numeral or year.

Valency states are represented by a superscript roman numeral, *e.g.*, Fe^{II}, Mo^{V}. Substances in the ionic state are represented by Na^+, Fe^{2+}, Fe^{3+}, etc., for cations and by Cl^-, SO_4^{2-}, PO_4^{3-}, etc., for anions.

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